n3

DOUGLAS & LOMASON COMPANY

Corporate Offices: 24600 Hallwood Court, Farmington Hills, MI 48335 - 1671 • *Fax (313) 478-5189*
Telephone (313) 478-7800

Please Reply to:
P.O. Box 20783, Atlanta Airport
Atlanta, Georgia 30320
Telephone (404) 349-7000

December 6, 1991

Mr. James V. Callier
Chief, IOWA Section
RCRA Branch/WSTM Division
US EPA - Region VII
726 Minnesota Avenue
Kansas City, Kansas 66101

Re: Closure Plan for Hazardous Waste Container Storage Area
Douglas & Lomason Company, Red Oak, Iowa
EPA ID No. IAD041107871

Dear Mr. Callier:

Please find two copies of the revised closure plan as requested in your letter dated September 12, 1991 and received in this office September 23, 1991.

The twenty items that were addressed in your letter were incorporated into the revised document as follows:

Item 1: Changed throughout plan
Item 2: Subsection 3.2.4.2
Item 3: Subsection 3.3
Item 4: Subsection 3.3
Item 5: Subsection 4.3
Item 6: Subsection 3.3
Item 7: Subsection 3.3
Item 8: Subsection 3.3
Item 9: Subsection 4.2 & Lab QA Plan
Item 10: Subsection 4.2.1
Item 11: Modified Table 4-1
Item 12: No item in letter
Item 13: Subsection 3.3
Item 14: Subsection 3.3.3
Item 15: Subsection 3.2.5
Item 16: Subsection 3.3.1
Item 17: Subsection 3.2.4.2
Item 18: Subsection 3.2.6
Item 19: Subsection 3.2.6
Item 20: Subsection 3.2.7

If additional information is needed, please contact me at (404) 349-7000.

Sincerely,

DOUGLAS & LOMASON COMPANY

Raymond L. Osborne
Corporate Environmental Manager

CC: Steve Warywoda, Plant Manager
Bob Stachura, Executive Manager & VP
Warren Daubenspeck, VP, Safety, Environmental & Loss Control

Drum Storage Area Closure Plan

DOUGLAS & LOMASON COMPANY

Red Oak, Iowa

December 1991

HDR Engineering, Inc.

TABLE OF CONTENTS

DRUM STORAGE AREA CLOSURE PLAN

DOUGLAS & LOMASON COMPANY RED OAK, IOWA FACILITY

1.0 Introduction

SECTION 1.0

INTRODUCTION

1.1 Purpose and Scope

The purpose of this document is to present a plan for closure of the drum storage area at the Douglas & Lomason Company (Douglas & Lomason), Red Oak, Iowa Facility (Red Oak) following the guidance set forth by the Resource Conservation and Recovery Act (RCRA), 40 CFR Part 265, Subpart G. Following completion of the closure activities, a final closure certification report will be prepared describing the closure activities undertaken, presenting and summarizing the results of those activities and certification of final closure of the drum storage area.

Section 2 briefly discusses the history of the Red Oak facility and a description of the drum storage area.

Section 3 presents the closure activities which address all applicable subparts, and paragraphs contained in 40 CFR Part 265, Subpart G.

Section 4 presents the rationale, procedures and protocols to be utilized during the site investigation. This site investigation will provide the information and data necessary to prepare the certificate of closure.

1.2 Closure Objectives

The objective of this closure activity is to certify that there are no analytes of concern, in excess of the regulatory levels as identified in later sections, present in the area of the facility previously designated for drum storage. HDR Engineering, Inc. (HDR) will document the sampling activities, analytical results and quality control procedures that provide for the certification of closure.

2.0 Background

SECTION 2.0

BACKGROUND

2.1 Site Description

The Douglas & Lomason's Red Oak facility is located at 2700 North Broadway, Red Oak, IA., approximately one quarter mile north of the intersection of Highways 34 and 48. The site is bounded on the north by a Burlington Northern rail spur and open field, on the east by open field, on the south by a parking area and another manufacturing facility not associated with Douglas & Lomason, and on the west by Highway 48, as illustrated in Figure 2-1.

Figure 2-2 illustrates the facility modifications that have taken place since 1981 when the drum storage area was operated.

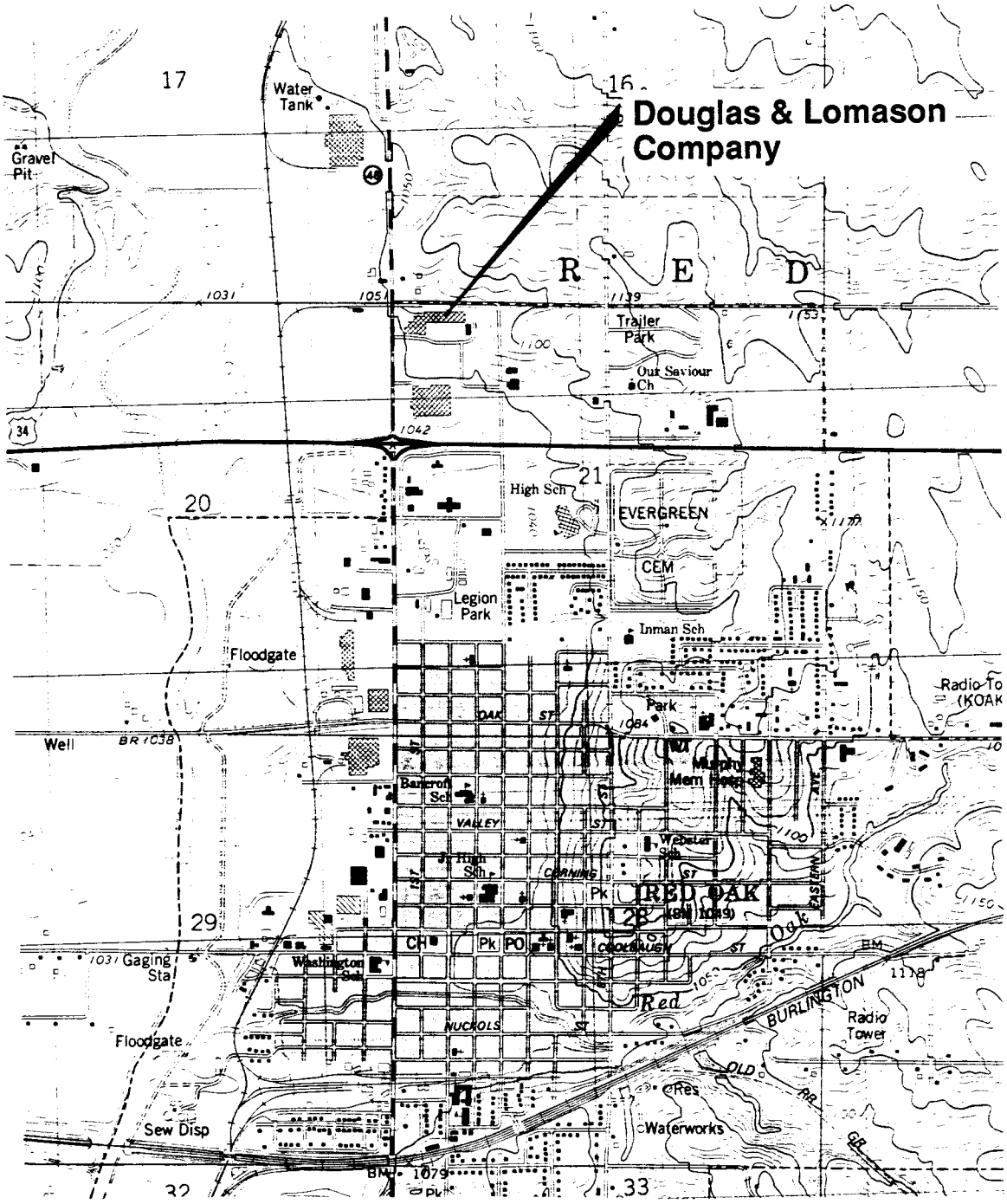
Figure 2-3 illustrates the exiting facility plan and indicates the location of the area inside of the current facility that was previously designated for drum storage. Figure 2-4 illustrates an approximate boundary location for the drum storage area located within the facility.

2.2 Facility History

In 1981, Douglas and Lomason applied for and received a Treatment/Storage/Disposal Facility (TSDF) interim status permit as regulated under RCRA for the drum storage area. The purpose of the drum storage area was established to provide secure storage of filter cake materials (dewatered sludges) containing chrome and zinc. Presented in Appendix A is a copy of the laboratory analysis for the filter cake. During this time the facility was configured such that the drum storage area was located inside of the building on a concrete slab. The drum storage area was under roof at all times and was bounded by concrete slab on all four sides. In 1982 the storage of drums in

N

Scale: 1" = 2000'



HDR

HDR Engineering, Inc.

Location Plan

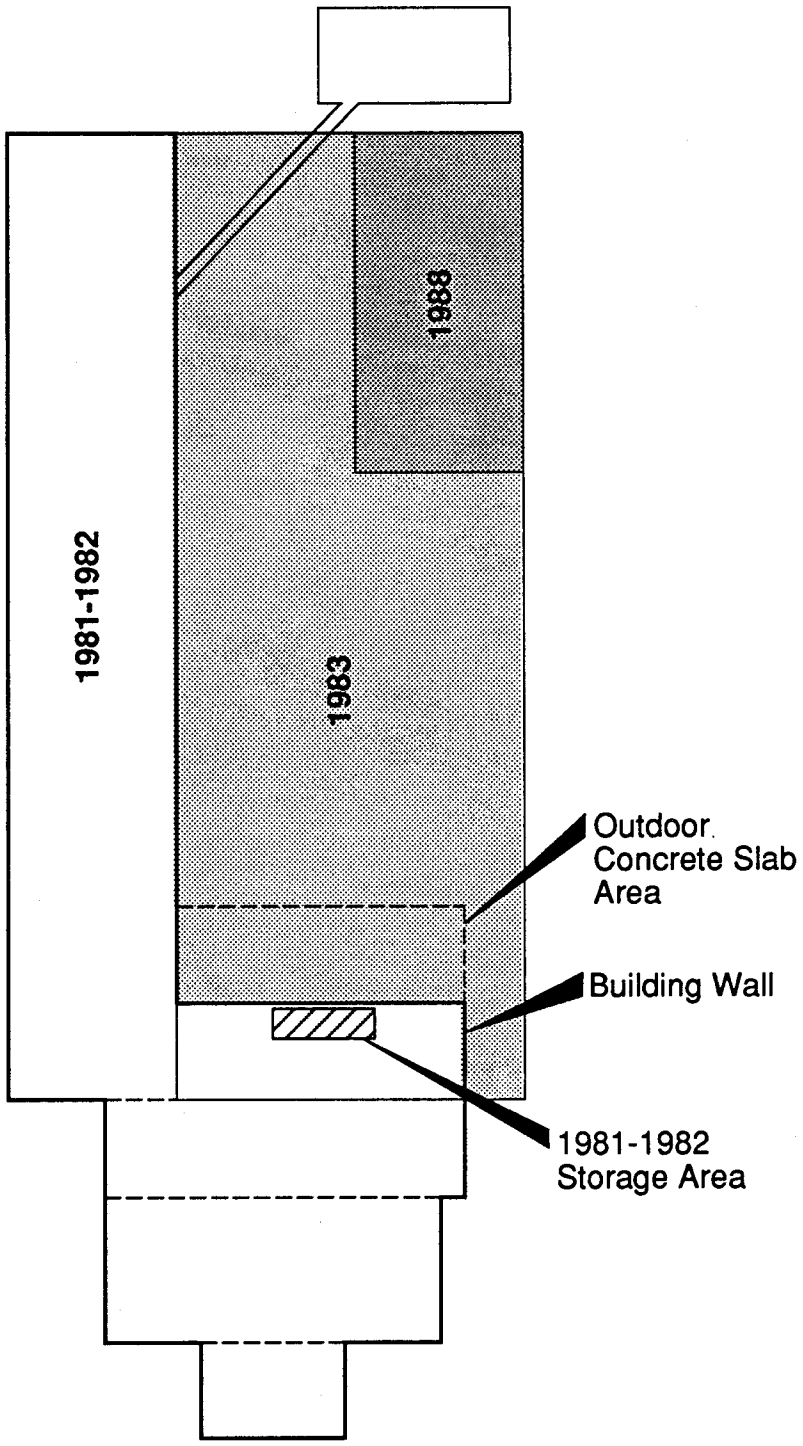
DOUGLAS & LOMASON COMPANY
Drum Storage Area
Closure Plan

Date
Nov. 1991

Figure
2-1

11-22-91
Location Plan
IES\Douglas & Lomason\Red Oak

No Scale



HDR Engineering, Inc.

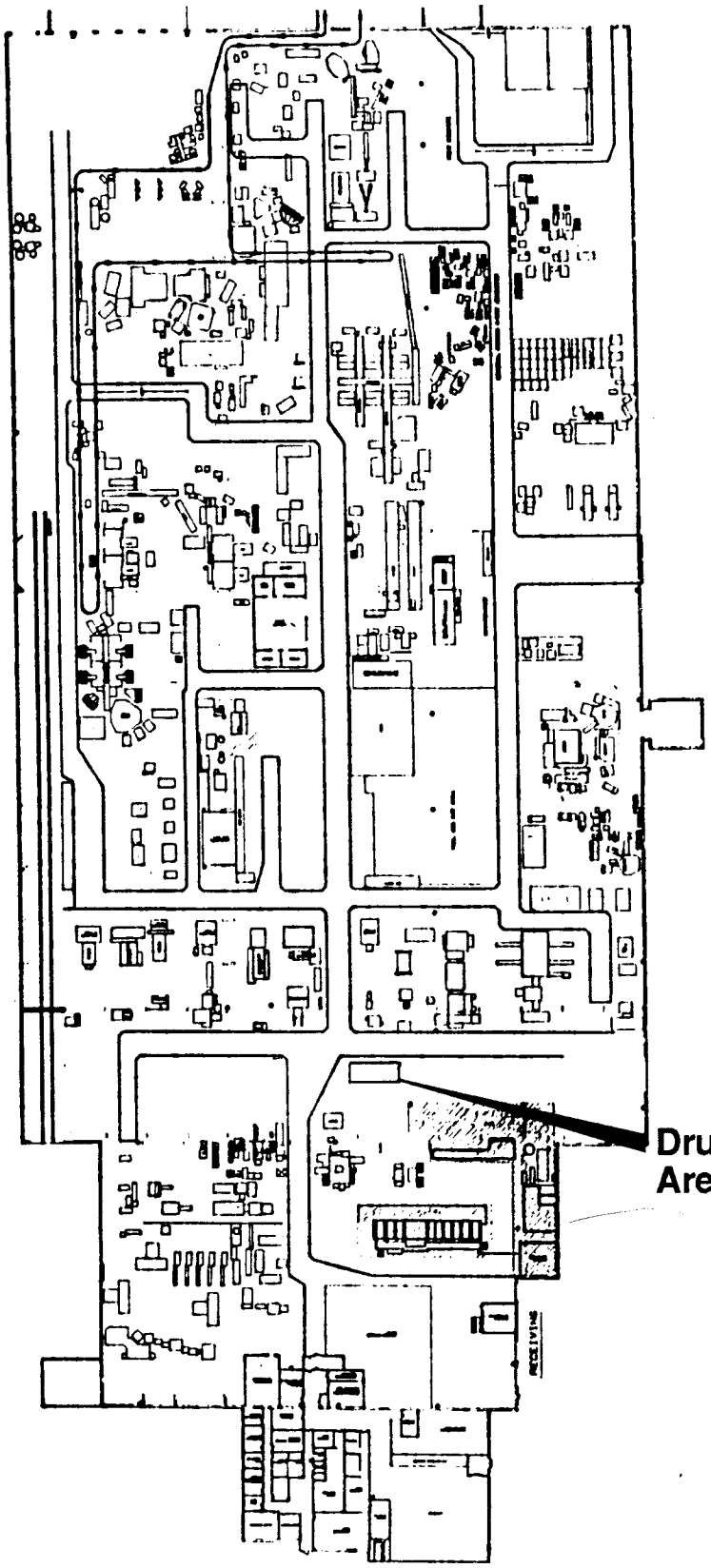
Facility Modification Plan

DOUGLAS & LOMASON COMPANY
Drum Storage Area
Closure Plan

Date
Nov. 1991

Figure
2-2

11-22-91
Facility Modification Plan
IES\Douglas & Lomason\Red Oak



Facility Plan

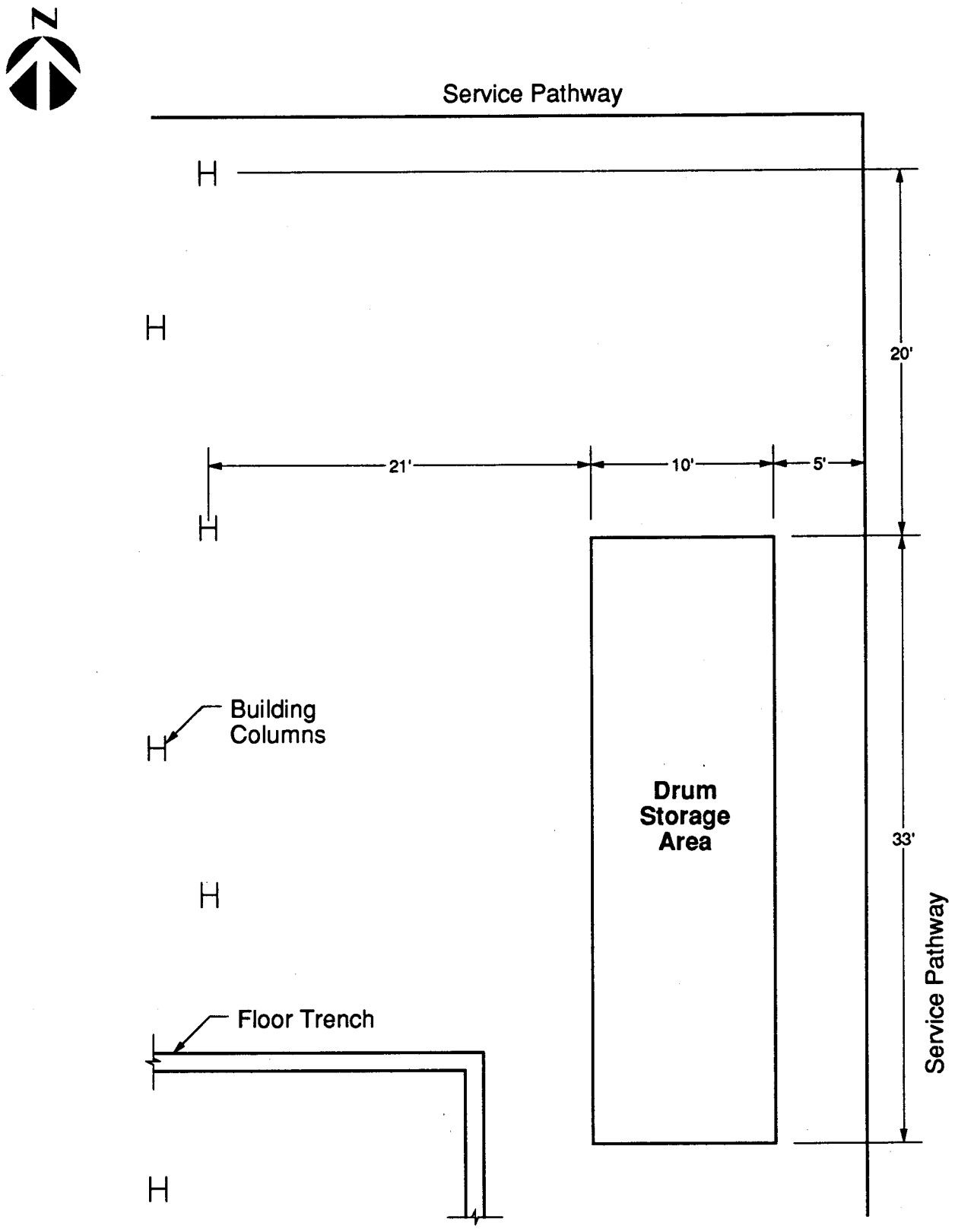
HDR Engineering, Inc.

DOUGLAS & LOMASON COMPANY
Drum Storage Area
Closure Plan

Date
Nov. 1991

Figure
2-3

11-22-91
Facility Plan
IES\Douglas & Lomason\Red Oak\Figures



HDR Engineering, Inc.

Drum Storage Plan

DOUGLAS & LOMASON COMPANY
Drum Storage Area
Closure Plan

Date
Nov. 1991

Figure
2-4

11-22-91
Drum Storage Area
IES\Douglas & Lomason\Red Oak\Figures

this storage area was discontinued. The building was expanded in 1983 and 1988 to approximately its present size as illustrated in Figure 2-2.

Past facility photos, visual identification of construction joints and interviews with existing personnel played key roles in determining the approximate location of the drum storage area.

3.0 Closure Activities

SECTION 3.0

CLOSURE ACTIVITIES

3.1 General

Federal regulations applicable to the Red Oak facility Drum Storage Area Closure are summarized in this section. The primary subpart which regulates the closure of this area is Subpart G, Closure and Post-Closure.

3.2 Federal Regulations

Applicable sections from the Code of Federal Regulations Title 40 (40 CFR), Protection of Environment, are summarized below.

3.2.1 40 CFR Part 265 Subpart G - Closure and Post-Closure

Sections 265.110 through 265.115 of Subpart G provides closure requirements which apply to all hazardous waste management facilities having interim status. The following is a listing of these sections and a brief explanation of each.

3.2.2 Applicability (265.110(a))

This plan, detailing the closure of the Douglas & Lomason Drum Storage Area, applies to owners and operators of all hazardous waste management facilities having interim status.

3.2.3 Closure Performance Standard (265.111)

Douglas & Lomason fully intends to close the Drum Storage Area in a manner that minimizes the need for further maintenance. At this time, it is not believed that it will be necessary to address the potential for post-closure escape of hazardous waste, hazardous constituents, leachate, contaminated run-off, or hazardous waste decomposition products to the ground or surface waters or to the atmosphere.

3.2.4 Closure Plan; Amendment of Plan (265.112)

The following subsection addresses the requirements of Section 265.112.

3.2.6 Certification of Closure (265.115)

Within sixty (60) days of completion of closure, Douglas & Lomason will submit to the Regional Administrator by registered mail a certification that the drum storage area has been closed in accordance with specifications of the approved closure plan. This certification will be signed by Douglas & Lomason and by an independent registered professional engineer. Included with the certification will be the supporting documentation describing the closure activities, sampling locations and analytical results, and chain of custody forms.

3.2.7 Closure Cost Estimate (265.142)

A cost estimate for final closure is presented below.

Site Sampling and Cleaning	$4,000
Site Restoration and Repair	$4,000
Analytical	$5,000
Engineering	$16,000
Subtotal	$29,000
Contingency (20%)	6,000
Total	$35,000

3.3 Closure Plan

Douglas & Lomason is planning to demonstrate that the existing structural floor slab in the area previously known as the Drum Storage Area does not contain elevated levels of analytes associated with the storage of filter cake material in drums. Furthermore, Douglas & Lomason plans to demonstrate, if it is deemed necessary during closure activities, that none of the subsoils beneath the drum storage area present a hazard to human health and the environment; and that no closure/post-closure escape of hazardous waste, hazardous constituents, leachate, contaminated run-off, or hazardous waste decomposition products to the ground or surface waters or to the atmosphere could occur.

This closure plan addresses the requirements of 40 CFR Sections 265.110 through 265.115.

The following paragraphs and Figure 3-1, Closure Activity Diagram, describe the activities to be performed during closure.

3.3.1 Inspection

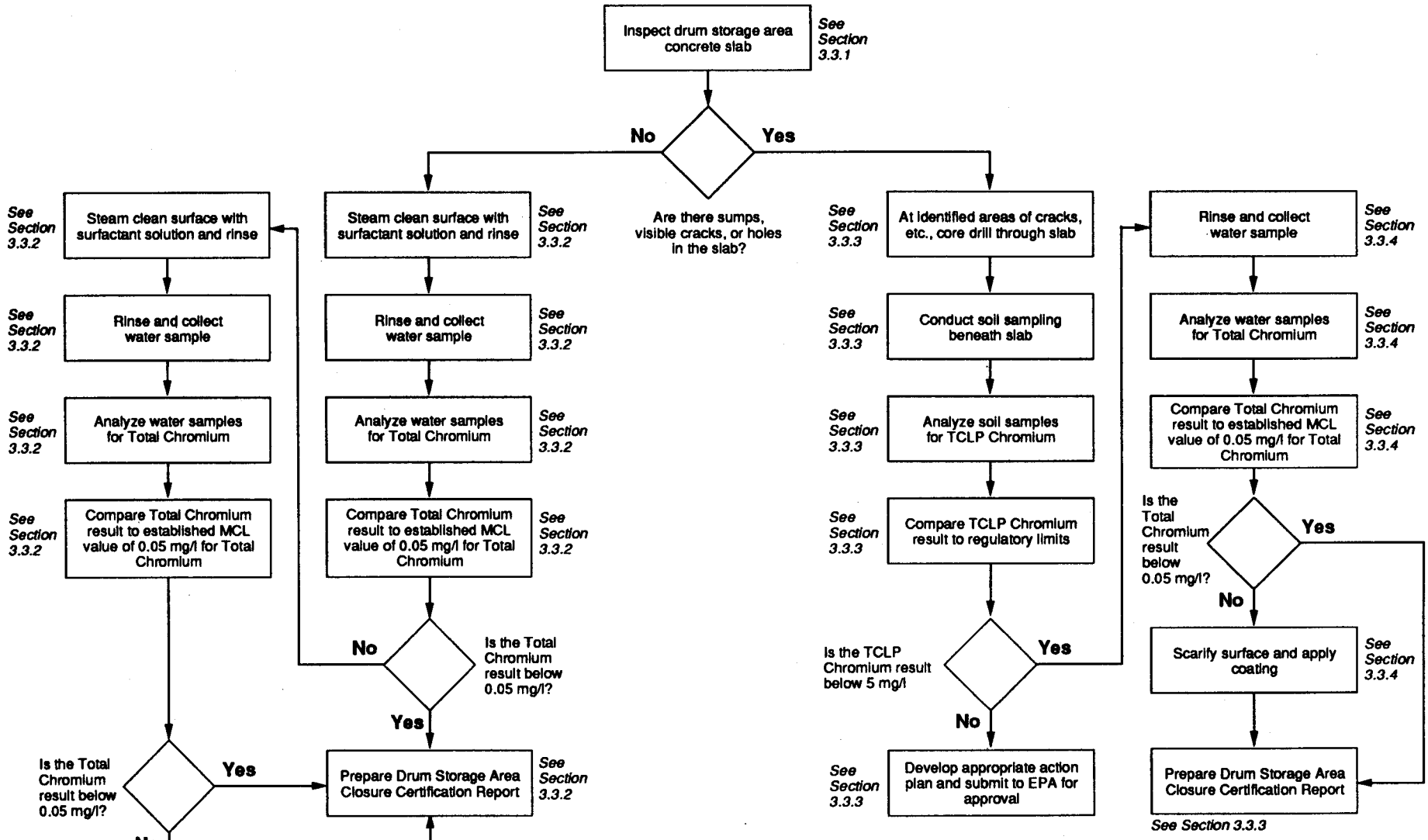
Initial site activities will involve the inspection of the concrete slab of the drum storage area. The concrete slab will be inspected for sumps, cracks, holes and crevices. HDR, the certifying engineer, will compare the observed conditions to a permissible crack or pathway width of 0.016 inches. This permissible crack width is as established by the American Concrete Institute (ACI) to protect reinforcing members from corrosion through intrusion of water.

If there are no pathways observed through the concrete slab greater than the permissible, action will be taken to close the drum storage area according to the procedures outlined in Subsection 3.3.2, Surface Cleaning, and in Figure 3-1.

If there are pathways observed through the slab greater than the permissible, action will be taken to identify the potential presence of analytes of concern both on top of the concrete slab and below the concrete slab at the location of the cracks or crevices. Procedures outlining the investigative steps below the concrete slab are presented in Subsection 3.3.3, Subsurface Investigation, and in Figure 3-1. Procedures outlining the investigative steps on top of the concrete slab are presented in Subsection 3.3.4, Surface Investigation, and in Figure 3-1.

It should be noted that Douglas & Lomason will notify the EPA in writing at least thirty (30) days prior to the planned sampling event(s) so that EPA personnel or a representative can, if necessary, be on-site during the sampling activities.

12-2-91
Closure Activity Diagram 3-1
IES\Douglas & Lomason\Red Oak\Figures



HDR Engineering, Inc.

Closure Activity Diagram

DOUGLAS & LOMASON COMPANY
Drum Storage Area
Closure Plan

Date
Dec. 1991

Figure
3-1

3.3.2 Surface Cleaning

If it is determined that there are no pathways in which the analytes of concern could potentially have migrated beneath the concrete slab, the top of the concrete slab will undergo the following steps in order to meet clean closure requirements.

Initially the drum storage are will be encircled with absorbent rings to contain the wash and rinse waters. The concrete slab will be steam cleaned with a wash solution consisting of a non-foaming surfactant and city water. The city water will be sampled and analyzed for total chromium. The wash solution will then be collected by a vacuum. The concrete slab will be rinsed with city water and collected by vacuum. The concrete slab would again be rinsed with city water and collected by vacuum. This rinsate would be sampled and analyzed for total chromium. See Subsection 4.2.1, Sampling Method, for the sampling method to be used. See Subsection 4.2.2, Analytical Method, for the analytical methods to be used.

The analytical results will be compared to the health-based drinking water standards promulgated as maximum contaminant levels (MCLs). These standards are presented in 40 CFR Subsection 141.11(a), Maximum Contaminant Levels for Inorganic Chemicals. The MCL for total chromium is 0.05 mg/l. If the analytical results for total chromium are below the MCL value, then HDR would prepare a closure certification report for the drum storage area. If the analytical results are above the MCL value, then the steam cleaning, rinsing and sampling process would be repeated. The analytical results from the second sampling event would be compared to the MCL value. If the analytical results for total chromium are below the MCL value, then HDR would prepare a closure certification report for the drum storage area. If the analytical results continue to be above the MCL value, then the surface of the drum storage area would be mechanically scarified a depth of approximately

0.25 inches. The scarified material would be collected, drummed, analyzed, and if necessary, manifested for shipment by a licensed hauler to a licensed landfill capable of accepting the waste material. The drum storage area would then be covered with an epoxy floor coating to seal the concrete and provide a smooth finished surface. Upon completion of the coating application, HDR would prepare a closure certification report for the drum storage area.

3.3.3 Subsurface Investigation

If it is determined by the certifying engineer that there are pathways in which the analytes of concern could potentially have migrated beneath the concrete slab, then a subsurface investigation of the underlying soils would be performed. Subsurface soil sampling will be performed at identified cracks with a frequency of one sample per crack. The sampling method for the subsurface soils is described in Subsection 4.2.1, Sampling Method. The soils would be sampled and analyzed for total chromium and Toxicity Characteristic Leaching Procedure (TCLP) chromium. See Subsection 4.2.2, Analytical Method, for the analytical methods to be used.

The analytical results will be compared to the regulatory limit for TCLP chromium of 5.0 mg/L. If the analytical results for TCLP chromium are below the TCLP regulatory limit, then HDR would conduct the surface investigation of the concrete slab. See Subsection 3.3.4, Surface Investigation, for procedures of the investigation. If the analytical results for TCLP are above the TCLP regulatory limit, then HDR would develop an appropriate action plan for further investigation and submit that document to EPA for review and approval. The top of the concrete slab would not be investigated at that time.

3.3.4 Surface Investigation

The top of the concrete slab will be investigated in a similar manner to that discussed in Subsection 3.3.2, Surface Cleaning. Initial steps will be to encircle not only the entire drum storage area but all cracks, holes, etc. with absorbent rings in order to prevent rinse water from penetrating through the pathway. The rinse water would be collected by a vacuum. See Subsection 4.2.1, Sampling Method, for the sampling method to be used. The rinse water would be analyzed for total chromium. See Subsection 4.2.2, Analytical Method, for the analytical method to be used.

The analytical results would be compared to the MCL for total chromium. If the analytical results for total chromium are below the MCL value, then HDR would prepare a closure certification report for the drum storage area. If the analytical results are above the MCL value, then the concrete surface would be mechanically scarified as previously discussed in Subsection 3.3.2, Subsurface Cleaning. Upon completion of the scarification process, HDR would prepare a closure certification report for the drum storage area.

4.0 Site Investigation

SECTION 4.0

SITE INVESTIGATION

4.1 General

Sampling for closure certification will be completed by an HDR Engineering, Inc. field team. The sampling will be conducted to determine whether analytes of concern are present on the concrete surface or potentially have migrated into the subsoil. This sampling program involves surface sampling of the concrete slab and, if determined necessary, concrete coring through the existing structural slab followed by soil sampling beneath the slab.

4.2 Sampling and Analytical Methods

The appropriate sampling technique was selected for this location based on a visual assessment of the sample location.

4.2.1 Sampling Method

Following the cleaning procedure outlined in Section 3.3.2, sampling of the rinse water will be obtained according to the following procedure:

- Remove top from wet vacuum and visually inspect sample and note observations in field log book.
- With the "COLIWASA" in the open position, slowly lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same.
- When the sampler stopper hits the bottom of the container, close the sampler and slowly withdraw the sampler with one hand while wiping the sampler tube with a disposable paper towel with the other hand.
- Carefully discharge the sample into a suitable sample container.
- Repeat above procedures until sufficient sample volume is obtained.

In the event it is determined subsurface soils will be sampled, the concrete slab will be cored with a coring machine fitted with a 4-inch inner

diameter bit. The concrete slab will be cored and the core extracted leaving an opening through which access to the underlying soils is gained. Soil samples will be obtained by hand driving a 2-inch nominal diameter split spoon sampler into the appropriate sampling interval. This method was chosen due to restrictions in terms of available space to get a drilling rig inside of the facility. Samples are to be obtained at the 0-8 inch, 8-16 inch and the 16-24 inch intervals at each coring location. If the above sampling intervals cannot be attained, alternate intervals will be determined in the field at the time of sampling. If insufficient recovery of a sample interval occurs, a sample from the previous recovered interval will be utilized as the sample point to be analyzed. The samples will be obtained according to the following procedures:

- Soil samples will be obtained from the appropriate depth interval by hand driving a split spoon sampler to the specified depths.
- Remove split spoon sampler, open, visibly inspect sample and note recovery and observations in field log book.
- The soil sample will be placed in a stainless steel mixing bowl.
- The soil will be mixed to obtain a homogeneous sample representative of the sample interval.
- Two 8-oz. glass containers will be filled. One will be sent to the laboratory for analyses, the other will be sent to the Douglas & Lomason Atlanta office for reference.

When the soil sampling at the coring locations has been completed, the borings will first be filled with a bentonite/grout mixture followed by a sand/gravel concrete mix to provide structural integrity.

4.2.2 Analytical Method

This section discusses the analytical methods to be used for the analysis of rinse water and soil samples. Analytical methods used by the laboratory are those detailed in EPA Methods Manual SW-846 dated November 1986, including any future revisions, and 40 CFR Part 261, Appendix 16 -

Method 1311 Toxicity Characteristic Leaching Procedure ("TCLP"). Any deviations from these methods will require the concurrence and approval of EPA Region VII and Ray Osborne, Corporate Environmental Manager, Douglas & Lomason. Analytical methods, sample containers, holding times, and preservatives are outlined in Table 4.1, Analytical Methodology.

PACE Laboratories, Inc. of Minneapolis, Minnesota is the analytical laboratory performing sample analysis. Presented in Appendix B is a copy of PACE Laboratories Quality Assurance Plan.

4.3 Decontamination Procedures

Decontamination is intended to minimize the potential for cross-contamination between samples. The sampling activities supporting this closure certification require the use of disposable and reusable equipment. Disposable or single-use equipment will be used to the greatest extent possible. Items such as stainless steel bowls, spoons, and concrete core bits are reusable and require decontamination. Decontamination procedures:

- Wash using non-foaming surfactant in city water.
- City water rinse.
- Distilled water rinse.
- Air dry.

Investigation-derived contaminated materials such as disposable gloves, disposable sampling equipment, tyvek coveralls, decontamination solutions, etc. require disposal. Concrete coring flush water and equipment decontamination solutions will be collected in a 55-gallon drum and left at the site for disposal by Douglas & Lomason. Remaining soil materials and cores will be placed in a 55-gallon drum and left at the site for disposal by Douglas & Lomason.

TABLE 4.1

Analytical Methodology

Matrix	Analyte	Analytical Method[1,2]	Container	Preservative	Holding Time
Rinse Water/City Water	Total Chromium	SW-846 Method 3010/6010	Plastic/Glass	HNO_3 pH < 2	6 months
Soil	Total Chromium	SW-846 Method 3050/6010	Plastic/Glass	Cool 4°C	6 months
	Toxicity Characteristic Leaching Procedure	Method 1311	Plastic/Glass	Cool 4°C	Extract as soon as possible
	TCLP Extract Total Chromium	SW-846 Method 3010/6010	Plastic/Glass	HNO_3 pH < 2, Cool 4°C	Analyze as soon as possible following extraction

1. Test Methods for Evaluating Solid Waste EPA SW-846, Third Edition, November 1986. Including any future revisions.
2. 40 CFR Part 261, Appendix 11 - Method 1311 Toxicity Characteristic Leaching Procedure (TCLP).

4.4 Field Quality Control Procedures

Background samples and duplicate samples are included in this sampling program as part of the quality control/quality assurance program. One background sample will be taken of the City water to determine the background levels of the total chromium. One duplicate sample will be collected. The laboratory performing the analyses will not be notified regarding which sample is the duplicate sample.

Other field quality control samples used include decontamination blanks.

On decontamination blank will be prepared with HPLC Grade Water by pouring water over the decontaminated split spoon sampler. The decontamination blank will be labeled, prepared, and packaged according to the sample preparation procedures.

Also, remaining rinse water left in the container will be drummed and left on site for disposal by Douglas & Lomason until analytical results are available.

4.5 Sample Preparation

The Site Coordinator will be responsible for sample handling. The Site Coordinator will check that the appropriate containers are used, the sample containers were decontaminated prior to shipment, the samples are preserved in the appropriate manner, each sample is properly identified, and the proper packaging and shipping methods are used.

4.5.1 Sample Containers

Containers will be appropriately sized and of the proper material to meet the analytical requirements. The containers will be pre-cleaned by the laboratory, in accordance with the analytical methods being used, prior to being shipped.

4.5.2 Sample Preservation

Field sampling personnel will preserve each sample collected for laboratory analysis according to the specified preservation requirements. The laboratory will provide the required preservatives as appropriate to this investigation.

4.5.3 Sample Container Decontamination

After collection and prior to leaving the site, the exterior of all sample containers will be decontaminated. The decontamination steps included at a minimum:

- City water rinse (sample container)
- Non-foaming surfactant and city water wash
- City water rinse

4.5.4 Sample Identification

Each sample will be assigned a unique sample identification number to allow for proper data management. The sample numbers will be included on the sample label and in the daily field log book to identify notes pertaining to the sample and on the chain-of-custody forms.

Samples will be labeled immediately after collection. Information included on the sample label:

- HDR sample identification number
- Sample type
- Date and time of collection
- Name of the sampler
- Sample collection location
- Requested analysis

The labels will be filled out in indelible ink, firmly affixed to the sample container and protected by covering with clear tape.

For simplicity and ease of tracking, the sample numbers will be assigned in sequential order. If, for any reason, a sample number is not used, an explanation of the reason will be included in the log book(s), and noted as UNUSED.

4.5.5 Sample Packaging

Labels on sample containers will be secured by wrapping with clear tape to prevent them from coming off during transportation. Each sample container will be placed inside the cooler and cushioning material added for stability during transportation. Ice packs and/or ice substitutes will be used to maintain a sample temperature of 4°C.

4.5.6 Sample Transportation

Chain-of-Custody sheets will be prepared for document control and transfer of samples from the field team to the analytical laboratory. The top two copies of the chain-of-custody record will be sealed in a polyethylene ziploc-type bag and taped to the inner lid of the cooler. The third (bottom) copy will be retained by the HDR Site Coordinator.

4.5.7 Chain-of-Custody Procedure

Written records of the sample handling will be kept each time the sample changes hands. Each person receiving custody of the sample is required to document the sample transfer on the HDR chain-of-custody records. The HDR chain-of-custody records have three carbon-type copies. The Site Coordinator will complete the records and send the top two copies, the original (white) and verification of sample delivery (yellow copy), along with the lab samples. The third copy (pink) will be retained by the Site Coordinator.

4.6 Documentation

The Site Coordinator will be responsible for the log book, sample tags, chain-of-custody records and correspondence. Following completion of field operations, documentation will be relinquished by the Site Coordinator to the HDR project manager for maintenance in the project records.

4.6.1 Daily Field Log

Daily field log entries will be made in abound book using indelible ink. Each page in the log book will be numbered, dated, and initialed. Entries include the following:

- Date and time of entry
- Purpose of sampling
- Name and address of field contact (federal, state, local)
- Sample number
- Depth of sample and location
- Date and time of sample collection
- Sample identification or explanatory notes
- References such as maps or photographs of sampling site
- Field observations
- Field measurements

4.6.2 Data Corrections

As previously stated, all data recorded in daily log books, sample identification labels, chain-of-custody records, and other forms will be written in indelible ink. These documents will be retained by the Project Manager. They will not be destroyed or thrown away, even if they are illegible, tattered, or contain inaccuracies that require a replacement document.

If an error is made on a document, corrections will be made by crossing a line through the error in such a manner that the original entry remained legible, and entered the corrected information. Corrections will be initialed and dated if different from the date of the original entry.

Appendix A

LANGSTON LABORATORIES, INC.

Laboratory Report

Date Received: November 19, 1981
Time Received: 3:20 pm
Date Completed: December 18, 1981

Submitted by: Douglas & Lomason
24600 Hallwood Court
Farmington Hills, MI 48018
Attn: Mr. S. D. Cramer
P. O. No.: R 03079

LLI Project No.: 81-7558

Sample Description: Sludge

Sample Identification	Analysis	Results
Filter Cake from Red Oak, IA	pH	8.1
	Total Solids	33.8%
	Total Cyanide	< 1 mg/kg
	Free Cyanide	< 1 mg/kg
	Specific Gravity	1.325 g/ml
	Total Sulfide	2,050 mg/kg
	BTU	< 1,000 BTU/lb
	Ash	27.4%
	Arsenic	< 0.9 mg/kg
	Barium	43 mg/kg
	Cadmium	3.4 mg/kg
	Chromium	2.6%
	Lead	42 mg/kg
	Mercury	0.065 mg/kg
	Selenium	< 64 mg/kg
	Silver	< 2 mg/kg
	Copper	66 mg/kg
	Zinc	5.1%
	Nickel	13 mg/kg

Comments:

EP Toxicity - PAGE 2

5 Apr 90 Rec'd from D&L during onsite meeting

Approved: [signature]
Alan Kerschen
Laboratory Director

2005 West 103rd Terrace Leawood, KS 66206 913/341-7800

Sample Description: Sludge

Sample Identification	Analysis	Results
Filter Cake from Red Oak, IA	EP Toxicity	
	Cadmium	0.076 mg/liter
	Chromium	196 mg/liter
	Lead	0.40 mg/liter
	Selenium	< 0.05 mg/liter

Appendix B

LABORATORY
QUALITY ASSURANCE
PLAN

PACE, Inc.

Submitted by:

_____

Approved by:

Roger C. Splinter, Ph.D.
Vice President

Rev. #0 - DATE: November 1, 1989
Rev. #1 - DATE: May 17, 1990
Rev. #2 - DATE: August, 1991

II. TABLE OF CONTENTS

LIST OF EXHIBITS

LIST OF TABLES

III. INTRODUCTION, PROGRAM OBJECTIVES, AND STATEMENT OF POLICY

A. INTRODUCTION

This Generic Quality Assurance (QA) Plan is written in compliance with the elements required in the U.S. EPA, "Guidelines and Specifications for Preparing Quality Assurance Program Plans." (QAMS-004 80, September 20, 1980). This document contains the required elements of a Quality Assurance Plan and is prepared in such a way that entire sections can be referenced in subsequent specific project plans. This Laboratory QA Manual defines the systems of quality control and quality assessment that constitute the comprehensive Quality Assurance Program at PACE, Inc. Quality Control consists of specific procedures applied to all phases of analysis from sample receipt through the final reporting of results. The purpose of quality control is to insure that quality goals are met under routine operating procedures. Quality assessment involves the continuous evaluation of data and monitoring of analytical processes for the purpose of insuring that the quality control systems are performing effectively.

B. PROGRAM OBJECTIVES

The major elements of the overall Quality Assurance Program are summarized below:

- Use of appropriate methodologies by technically competent, well-trained personnel with state-of-the-art instrumentation and equipment.
- Adherence to well-defined standard operating procedures with emphasis on good laboratory and measurement practices.
- Analysis and assessment of quality control samples including (but not limited to) matrix spike samples, duplicate samples, surrogate spikes, blanks, and independent laboratory control standards.
- Participation in external quality evaluation programs such as the EPA Water Pollution and Water Supply (WP & WS) Study Programs.
- Maintainance of accreditation by State, Federal, and other applicable agencies for work performed.
- Monitoring of internal and external compliance to procedures and assessment of the performance of the analytical methods.

C. STATEMENT OF POLICY

PACE, Inc. is committed to the policy of providing the highest quality product to its client. The validity and reliability of the information generated is maximized by the adherence to documented quality control procedures and quality assurance protocols. PACE emphasizes the application of sound quality assurance/quality control principles beginning with the initial planning of the project, through all the field and laboratory activities and ultimately to the generation of the final report. The principles of data quality objectives, representativeness, completeness, comparability, precision and accuracy are applied.

PACE is committed to providing the resources, including facilities, equipment and personnel, to ensure the adherence to rigorous QA/QC protocols. Individual Quality Assurance Project Plans are developed for monitoring analytical projects to conform with the established QA/QC protocols.

IV. LABORATORY ORGANIZATION AND RESPONSIBILITY

The organizational structures for PACE, Inc. are provided in Exhibits 1, 2, and 3.

Exhibit #1	Illustrates the PACE, Inc. Organizational Structure
Exhibit #2	Illustrates the PACE Corporate Structure with Regional Designation
Exhibit #3	Illustrates the full model on which all regional office structures are based

Job descriptions are provided within Quality Assurance Project Plans, as they are designed and developed to address specific projects.

EXHIBIT 1

PACE Organizational Structure

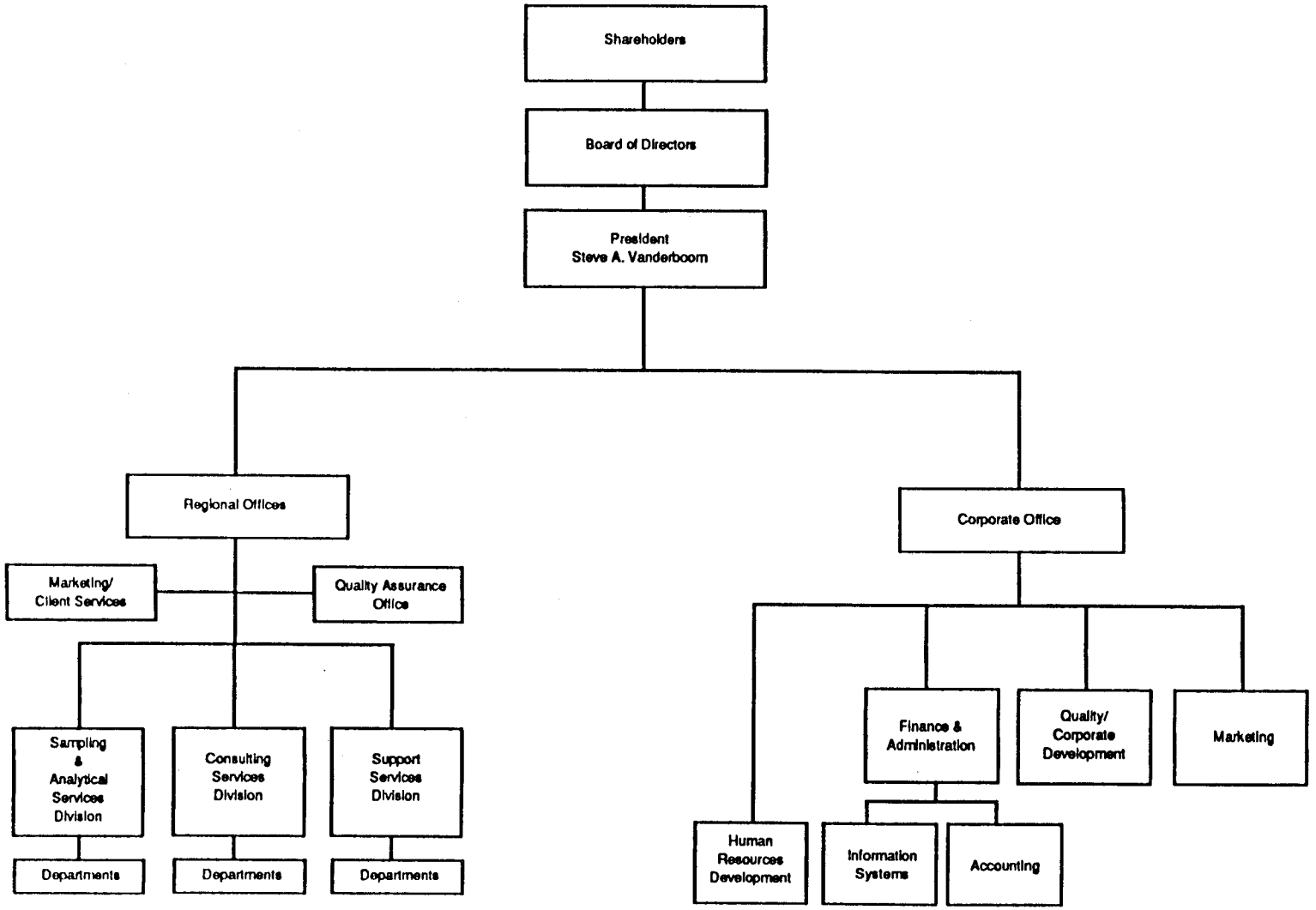
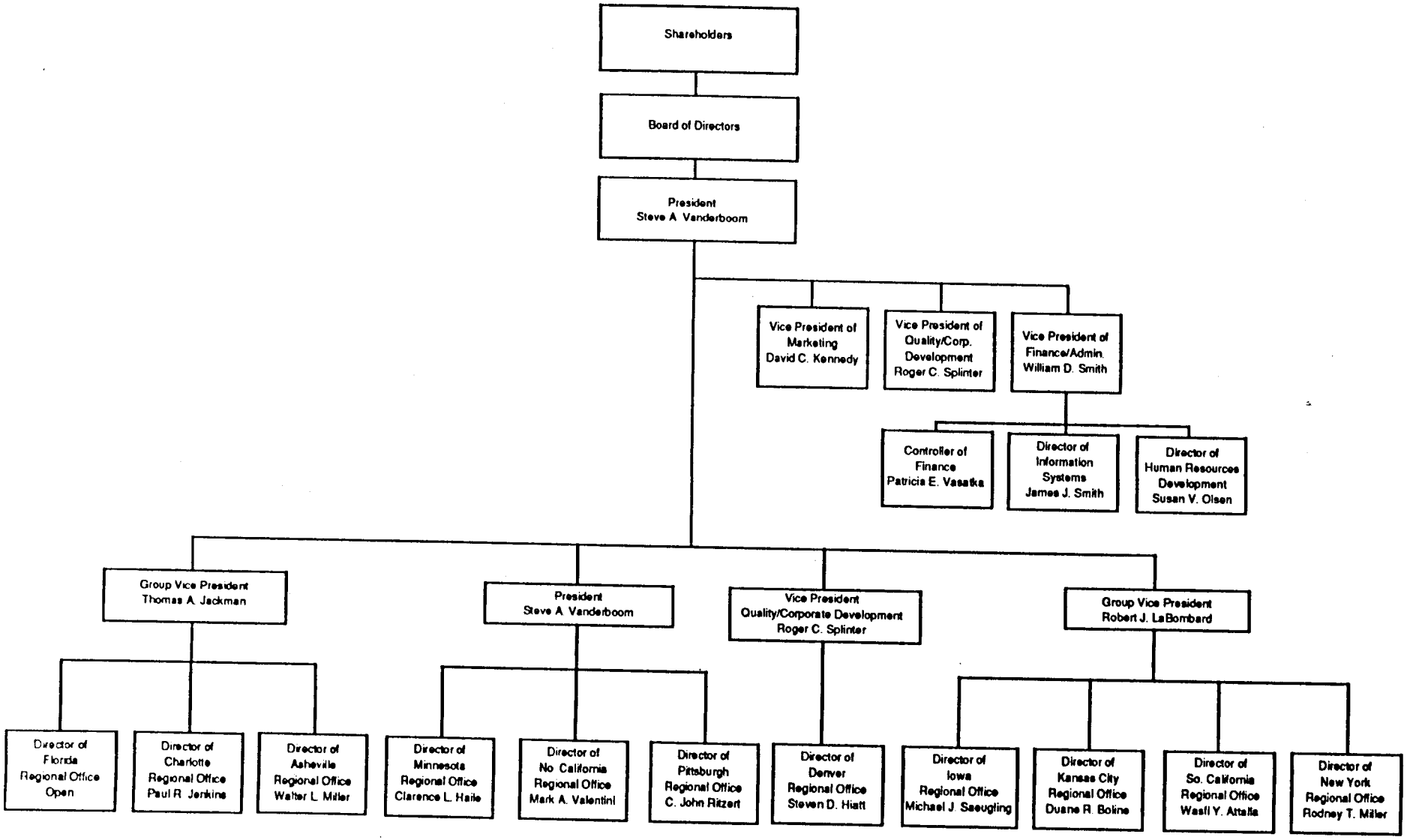


EXHIBIT 2

Corporate Management Personnel & Regional Directors

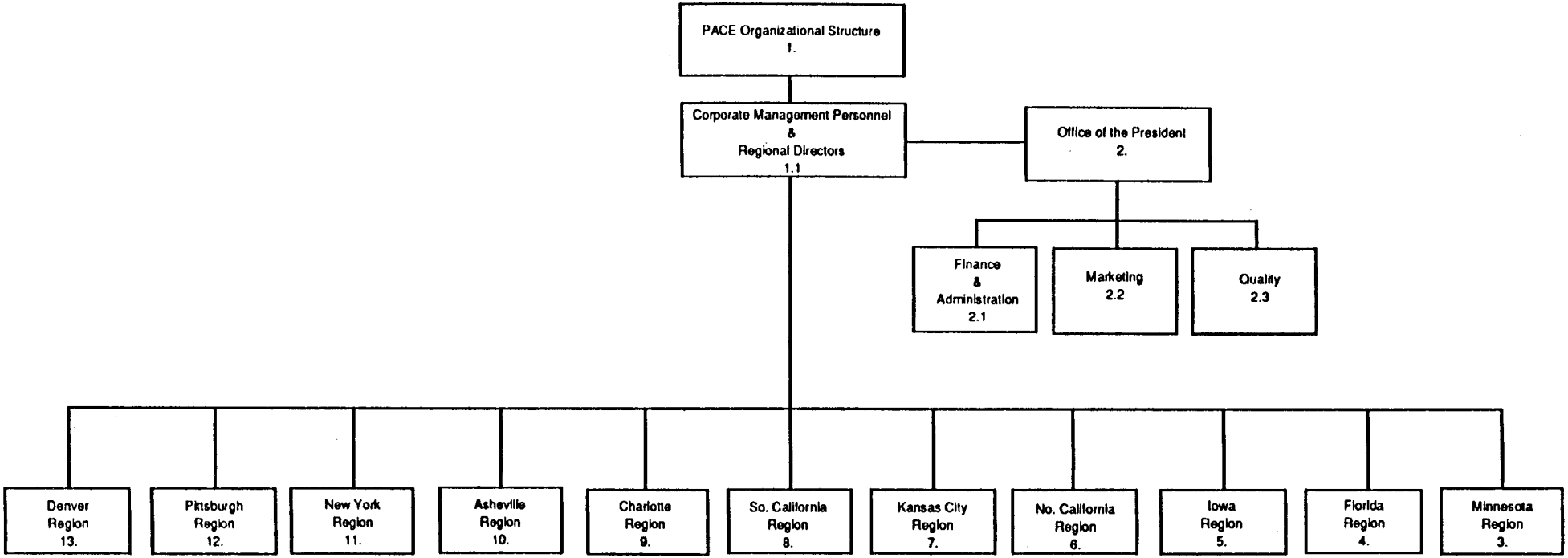
1.1



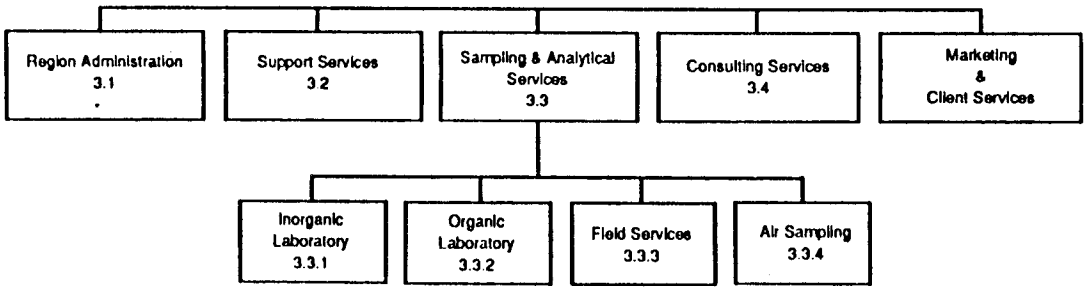
September 1991

EXHIBIT 3

Guide To Organizational Charts



These charts detail the MN Region divisions and departments and represent the full model on which all regional office structures are based.



August 1991

V. QUALITY ASSURANCE OBJECTIVES

The purpose of the plan is to define procedures for the documentation, evaluation, validation, and reporting of data. The objective is to provide a uniform basis for sampling, sample handling, instrument maintenance and calibration, methods control, performance evaluation and analytical data generation and reporting. Specific procedures to be used for sampling, chain of custody, calibration of field instruments (pH, conductivity meters, etc.), laboratory analysis, reporting, internal quality control, audits, preventive maintenance, and corrective actions are described in specific sections of this plan. This section addresses the objectives of accuracy, precision, completeness, representativeness, and comparability.

The QA objectives for precision and accuracy are to achieve the QC acceptance criteria specified in the proposed analytical procedures. For the organic and inorganic procedures, the precision and accuracy guideline requirements are specified in the individual methods.

Field blanks and duplicates are collected and analyzed to assess field sampling activities. The results check procedural contamination and/or ambient conditions at the site.

Due to the extensive number of organic parameters and potential matrices, the development of precision and accuracy objectives and control limits for every matrix is difficult. This is typically done with (1) matrix spike and matrix spike duplicate compounds which are added to selected samples before extraction and analysis, and/or (2) surrogate spike compounds which are added to every sample, before extraction and analysis. Although the surrogate and matrix spike analyses do not provide statistically valid statements about precision and accuracy for every compound in a sample, they do give the data reviewer enough information to make judgements about precision and accuracy on a sample-by-sample basis.

Inorganic precision and accuracy data are determined by using duplicate samples (precision), matrix spike and laboratory control samples (accuracy). The following procedure is used:

For a duplicate sample analysis, at least one duplicate sample is analyzed per sample matrix type (e.g. water, soil) and concentration (e.g. low, medium) per batch of samples or for each 20 samples received, whichever is more frequent, or as specified by state/project requirements. Samples identified as field blanks can NOT be used for duplicate samples analyses. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (i.e., ICP, GFAA), duplicate samples will be run by each method. The relative percent difference (RPD) for each component is calculated for later use during data assessment.

Completeness is a measure of all information necessary for a valid scientific study. For completeness, it is expected that the methodology proposed for chemical characterization of the samples collected will provide data meeting QC acceptance criteria following standard laboratory data review and validation for at least 95% of all samples collected. Completeness may also be defined as a comparison of the number of tests successfully completed (with acceptable QC) to the number of tests requested. Discrepancy reports are completed to provide explanation when QC criteria are not met.

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in each work plan.

Comparability is also considered during preparation of the work plan. The objective of comparability is to ensure that results of similar activities conducted by different parties are comparable. PACE uses EPA-approved or other methods and procedures to ensure comparability with data from previous or following studies. PACE participates in external and interlaboratory performance evaluation (PE) studies as additional means of establishing comparability in the laboratory.

VI. SAMPLING PROCEDURES

PACE, Inc. receives samples collected by clients and also has the capability to perform sampling for clients. PACE prepares or purchases sample containers in accordance with EPA-issued guidelines for container and preservative requirements. Pre-cleaned containers are obtained from reputable vendors and preservatives are added as required. Technical assistance from all supervisory and management staff is available to clients if needed.

A. BOTTLE PREPARATION PROCEDURES

The following is the procedure used for Sample Container Preparation:

1. Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide clear, consistent methods for preparing containers for sample collection. Following this procedure will facilitate accurate and consistent analytical results.

2. Application

The policies and procedures contained in this SOP are applicable to the personnel in the container preparation area.

3. General Policies

a. Always use new bottles when preparing containers for sampling (exception: One gallon, amber glass bottles used for transporting deionized water can be re-used after proper cleaning). These may be commercially-obtained precleaned bottles.

b. Always wear disposable latex gloves when handling sample containers.

c. Several preparation procedures require the use of acids as a preservative or cleaning agent.

   1. Be extremely careful when working with acids.

   2. Always wear safety glasses and a laboratory coat.

d. Bottle labels will list the preservatives added and the analysis to be performed, minimizing the probability for error.

e. When shipping pre-preserved bottles containing corrosives or oxidizers, consult proper DOT regulations.

4. **Procedures: Containers for Aqueous Samples**

a. **Volatile Organic (VOA) Sample Container Preparation**

1. Vial cleaning procedures.

a. Wash an entire package of vials in one washing session. Never store open packages of vials.

b. Soak the vials in cleaning solution (one capful of Acationox detergent, or equivalent, per sink of hot tap water) for 5 minutes.

c. After soaking, thrice rinse each vial thoroughly with hot tap water.

d. Thrice rinse each vial thoroughly with carbon filtered, deionized water (CFDI).

e. Stack rinsed vials in a drying tray (metal tray lined with aluminum foil, dull side exposed).

f. Bake the vials at 103°C for a minimum of four hours.

g. Cover baked vials with aluminum foil such that the dull side of the foil is in contact with the vials and set trays on a lab bench to cool.

2. Septum and cap cleaning procedures.

a. Clean entire packages of caps and septa. Do not store open bags.

b. Clean caps and septa separately.

c. The same procedures used for vial cleaning are used for cap and septum cleaning. Follow B through D in Section 1.

d. Spread evenly and thinly in drying trays to facilitate drying.

e. Dry for one hour at 103°C. Extended periods of heat can damage caps and septa.

f. Place clean caps and septa into a 1500 mL glass container which has been cleaned.

3. Assembling VOA vials.

   a. Place ten clean vials upright in a vial box with dividers. Recover drying trays with foil after vials have been removed.

   b. Add 4 drops of concentrated hydrochloric acid (HCL).

   c. Add (10 mg/40 ml) 0.008% sodium thiosulfate if chlorine is present (e.g. drinking water).

   d. Assemble a cap by inserting a septum in the cap such that the Teflon (white) side is exposed to the interior of the vial.

   e. Cap each vial tightly.

   f. Repeat assembly procedures until all vials are capped.

b. **Semivolatile Container Preparation**

1. Glass, amber jars (250, 500, and 1000 mL) with Teflon lined caps are used to hold samples for semivolatile analysis.

2. Bottles and cap liners are rinsed with reagent grade acetone. (Acetone is a target compound for some EPA methodologies and a CLP compound. If acetone interferes with the analyses, use of hexane and/or methanol may be an alternative, as specified in the method.)

   a. Acetone is highly flammable and acetone vapors are toxic.

   b. When using acetone, wear latex gloves, safety glasses and work in a vented hood.

   c. Pour a small amount of reagent grade acetone in the bottle to be rinsed.

   d. Cap the bottle with a Teflon lined cap.

   e. Shake the bottle making sure the acetone comes in contact with all sides of the bottle and the cap liner.

   f. Empty the bottle, invert it on a drying rack and allow it to air dry.

   g. Cap the bottle with a rinsed cap.

   h. Attach a blue dot to the top of the cap indicating the container has been acetone rinsed.

c. **Metals Container Preparation**

1. Polyethylene bottles (125, 250, 500, and 1000 mL) with plastic caps are used to hold water samples to be analyzed for metals.

2. Add a small amount of 1:1 nitric acid to a bottle.

3. Cap the bottle and shake vigorously, being certain the acid comes in contact with all interior surfaces.

4. Empty the container.

5. Rinse the bottle and cap thrice with deionized water.

6. Add the appropriate amount of 1:1 nitric acid, cap, and place a red dot on the cap to indicate the container contains nitric acid preservative.

Container Size	Quantity 1:1 Nitric Acid
125 mL	0.25 mL
250 mL	0.38 - 0.5 mL
500 mL	0.75 - 1.00 mL
1000 mL	1.5 - 2.0 mL

d. **Nutrient Container Preparation**

1. Polyethylene bottles (250, 500, and 1000 mL) with plastic caps are used to hold water samples for nutrient analysis.

2. Add the appropriate amount of sulfuric acid, diluted 1:1 from concentrate with carbon filtered deionized water, to each container.

Container Size	Quantity 1:1 Sulfuric Acid
250 mL	0.38 - 0.5 mL
500 mL	0.75 - 1.00 mL
1000 mL	1.5 - 2.0 mL

3. Attach an orange dot sticker to the cap of each prepared container.

e. **Cyanide Container Preparation**

1. Polyethylene containers (1000 mL) with plastic caps are used to hold samples for cyanide analysis.

2. Add one gram (8 to 10 pellets) or concentrated solution (1.5-2.0 ml 6N) of sodium hydroxide and one gram of ascorbic acid to each container. If chlorine is present in the sample, use ascorbic acid.

3. Attach a silver dot sticker to the cap of each prepared container.

4. Cyanide containers have a short shelf life; do not prepare in large quantities. (See #6b)

5. Due to a short shelf life, cyanide containers should be prepared as needed.

f. **Phenol Container Preparation**

1. Clear or amber glass, small mouth containers (1000 mL) with "poly seal" caps are used to hold samples for phenol analysis.

2. Add 1.5 - 2.0 mL of sulfuric acid, diluted 1:1 from concentrate with carbon-filtered deionized water, to each container.

3. Attach an orange dot sticker to the cap of each prepared container.

g. **Oil and Grease Container Preparation**

1. Clear or amber glass, wide-mouth containers (1500 mL) with foil lined caps are used to hold samples for oil and grease analysis.

2. 1000 mL amber glass containers with Teflon lined caps are acceptable.

3. Add five mL of 1:1 sulfuric acid to each container.

4. Attach an orange (color-coded) dot sticker to the cap of each prepared container.

h. **Sulfide Container Preparation**

1. Polyethylene bottles (250 mL) with plastic caps are used to hold samples for sulfide analysis.

2. Add 0.5 mL of zinc acetate and NaOH (to pH greater than 9) to each container.

3. Attach a white dot sticker to the lid of each prepared container.

i. **Total Organic Carbon (TOC) Container Preparation**

1. Polyethylene bottles (250 mL) with plastic caps are used to hold samples for TOC analyses.

2. Add 0.25 mL of 1:1 sulfuric acid.

3. Attach an orange dot sticker to each prepared container.

j. **Radiological Containers Preparation**

1. Polyethylene bottles (one gallon) with wax coated, paper lined caps are used to hold samples for radiological analysis.

2. Add five mL of 1:1 nitric acid to each bottle.

3. Attach a pink dot sticker to the cap of each prepared container.

k. **Carbon-Free Deionized (CFDI) Water Container Preparation**

1. One gallon, small-mouth, amber glass bottles with Teflon lined caps are used to transport CFDI water.

2. These containers can be reused after appropriate cleaning.

3. Wash the bottle in hot tap water and Acationox detergent, or equivalent, (American Scientific Products).

4. Thrice rinse the bottle with hot tap water.

5. Thrice rinse with CFDI water.

6. Bake the bottle at 103° until dry (at least four hours).

7. Remove the bottle from the oven, cover the mouth with foil, and let cool.

8. Cap the bottle with a new, Teflon lined cap.

1. **Other Container Preparation**

1. Polyethylene bottles (125, 250, 500, and 1000 mL) with plastic caps are used to hold samples for general chemistry analysis.

2. Clear glass bottles (125, 500, and 1000 mL) with foil lined caps are used to hold samples with high oil content to be analyzed for general chemistry parameters.

3. Amber glass, small neck bottles (500 mL) with Teflon-lined caps are used to hold samples for total organic halide (TOX) analysis.

5. **Procedure: Containers for Soil Samples**

a. **Volatile Organic Analysis Sample Container Preparation for Soil Samples**

1. Wide-mouth, amber or clear glass containers (65 mL-125 mL) with Teflon-lined caps are used to hold samples for volatile organic analysis.

2. The same preparations procedure is used as is used in preparation of VOA containers for aqueous samples except no preservative is added to the containers. (See #4a)

b. **Semivolatile Container Preparation**

1. Wide-mouth, amber glass jars (250, 500, and 1000 mL) with Teflon-lined caps are used to hold samples for semivolatile analysis.

2. Preparation procedures are identified as those used in preparation of semivolatile containers for aqueous samples. (See #4b)

c. **Inorganic Container Preparation**

1. Polyethylene bottles (125, 250, 500, and 1000 mL) with plastic caps or wide-mouth clean glass jars (4 oz., 8 oz., or 32 oz.) with teflon-lined caps are used to hold samples for inorganic analysis.

2. If the samples contain a large quantity of oil, clear glass jars (125, 500, and 1000 mL) with foil lined caps are used instead of the polyethylene bottles.

3. Container preparation procedures are identical to those used in preparation of general containers for aqueous samples.

6. Sample Container Quality Control and Lot Assignment

a. Bottles of a given type, prepared in one session, constitute a lot.

b. Lot sizes will vary, depending on the demand for a given bottle type.

c. When a lot is prepared, it is assigned an eight character lot code.

1. The first two characters indicate the bottle type.

GN: General Unpreserved
MU: Metals Unfiltered
NT: Nutrients
CN: Cyanide
PH: Phenol
OG: Oil and Grease
SD: Sulfide
GV: GC VOA Water
GC: GC VOA Solid
GL: GC Q-Amber
GS: GC Sm Amber
GM: GC Misc. Refrigerated
HW: Hazardous Waste
OC: Total Organic Carbon
OX: Total Organic Halides
RA: Radiological

A complete listing of codes can be found in Section I of the LDMS User's Manual (Project & Sample Data Entry)

2. The next three digits indicate the bottle size.

125: 125 mL
250: 250 mL
500: 500 mL
000: 1000 mL and one gallon

3. The last three digits are the lot number. They are assigned in sequential order.

4. When the lot code is assigned, it is documented appropriately.

5. The person who prepared the containers initials the Lot Sheet next to the lot code.

One container per lot (or at minimum frequency of 1%) is used to hold a deionized water blank. (ASTM type II) This blank is analyzed to determine the level of contamination in the lot.

- The appropriate analyses are performed for the given container type.
- Use carbon-filtered, deionized water for all blanks.
- Fill all containers, except VOA's, up to the neck of the bottle.
- Fill VOA's such that no bubbles are trapped when the vial is capped.
- Label each blank with the following information:

  Client: PACE, QC

  Sample description: (Lot Code)
  Date Collected:
  Collected by: (Initials)
  Time Collected:
  Analysis: (As indicated for the bottle type)
  Preservative: (Check appropriate preparation)

6. Complete a Chain-of-Custody form to accompany the samples. Client, sample description, time sampled, preservative, analysis: as listed on the bottle label.

   Report to: (Name of container preparation person)

   Project Name: Container QA

   Requested Due Date: Priority 2

   Matrix: H_2O

   Route samples and Chain-of-Custody to Sample Check-in.

7. **Sample Analysis Data Entry Form Tracking for Bottle Prep QC**

Forms will be kept in an Outstanding QC file.

a. When a Report of Laboratory Analysis is received for the project, the Sample Analysis Data Entry Form is moved to the Complete QC file.

b. A copy of the Report of Laboratory Analysis is then routed to QC Data Entry and data are entered into the appropriate data base.

c. The data are reviewed by the supervisor of the Bottle Preparation Area and signed off as being certified "clean" if the following criteria are met. All laboratory contaminants shall be at or below the stated detection limit. If this criterion is not met, the bottles are re-cleaned or discarded and another blank analyzed. If criteria are not met, the supervisor of bottle prep works with QA department to discover and rectify the problem in cleaning procedures.

Sample containers, preservatives, and holding times for representative analytical groups are listed in Table 1. Refer to 40CFR 136 for complete information and details.

pace laboratories, inc.

Inorganic Analytical Guide

TABLE 1

Common Non-Metals Analysis

Parameter	Typical Method(s)	Comparable SW-846 Method(s), If Applicable	Sample Container/ Preservative*	Preferred Volume (ml)*	EPA Holding Time*
Acidity	EPA 305.1		P, G/4°C	100	14 Days
Alkalinity	EPA 310.1/310.2		P, G/4°C	100	14 Days
Bacteria, Total Coliform	Standard Method 909A	9131/9132	WK/4°C	100	6 Hours
Bacteria, Fecal Coliform	Standard Method 909C		WK/4°C	100	6 Hours
Bacteria, Total Plate	Standard Method 907		WK/4°C	100	48 Hours
BOD, 5 Day	EPA 405.1		P, G/4°C	500	48 Hours
BOD, 5 Day Carbonaceous	EPA 405.1		P, G/4°C	500	48 Hours
Boron	EPA 212.3		HNO_3< 2	100	6 Months
Bromide	EPA 320.1		P, G	200	28 Days
COD	EPA 410.1/410.2		P, G/4°C, H_2SO_4	250	28 Days
Color	EPA 110.3		P, G/4°C	250	48 Hours
Chloride	EPA 325.2/325.3	9251/9252	P, G	100	28 Days
Chlorine, Residual	EPA 330.1		P, G	500	Immed.
Cyanide, Total	EPA 335.2	9010	P, G/4°C, NaOH pH > 12	500	14 Days
Fluoride, Total	Standard Method 413A		P	500	28 Days
Fluoride, Electrode	EPA 340.2		P	200	28 Days
Fluoride, (SPADNS)	EPA 340.1		P	500	28 Days
Grease & Oil	EPA 413.1	9070/9071	G/4°C, H_2SO_4	1500	28 Days
Haroness, Total ($CaCO_3$)	EPA 130.2		P, G/4°C	250	6 Months
Ion Chromatography (Including common anions such as: Br^-, Cl^-, F^-, NO_2^-, NO_3^-, $PO_4^{\equiv}$, $SO_4^=$, SO_3^-, & others)	EPA 300		P, G/4°C	100	48 hrs.
Nitrogen, Ammonia	EPA 350.1/350.2		P, G/4°C, H_2SO_4	500	28 Days
Nitrogen, Kjeldahl	EPA 351.2/351.3		P, G/4°C, H_2SO_4	1000	28 Days
Nitrogen, Nitrate	EPA 353.2	9200	P, G/4°C	100	48 Hours
Nitrogen, Nitrite	EPA 353.2		P, G/4°C	100	48 Hours
Nitrogen, Nitrate & Nitrite	EPA 353.2		P, G/4°C, H_2SO_4	100	28 Days
Nitrogen, Organic	EPA 351.3		P, G/4°C, H_2SO_4	100	28 Days
Odor	EPA 140.1		G/4°C	1000	24 Hours
Oxygen, Dissolved	EPA 360.1		G - Bottle & Top	500	Immed.
pH	EPA 150.1	9040/9041/9045	P, G/4°C	100	Immed.
Phenol	EPA 420.1	9065	G/4°C, H_2SO_4	1000	28 Days
Phosphorus, Total	EPA 365.1/365.2		P, G/4°C, H_2SO_4	100	28 Days
Phosphorus, Ortho	EPA 365.1/365.2		P, G/Filter	100	48 Hours
Silica, Dissolved	EPA 370.1		P/4°C	100	28 Days
Solids, Total	EPA 160.3		P, G/4°C	100	7 Days
Solids, Total Volatile	EPA 160.4		P, G/4°C	100	7 Days
Solids, Total Dissolved	EPA 160.1		P, G/4°C	100	7 Days
Solids, Total Suspended	EPA 160.2		P, G/4°C	100	7 Days
Solids, Suspended Volatile	Standard Method 209A		P, G/4°C	100	7 Days
Solids, Settleable	EPA 160.5		P, G/4°C	1 Gal.	48 Hours
Specific Conductance	EPA 120.1	9050	P, G/4°C	100	28 Days
Sulfate	EPA 375.4	9036/9038	P, G/4°C	100	28 Days
Sulfide, Total	EPA 376.1	9030	P, G/4°C, NaOH pH > 9, Zn acetate	500	7 Days
Sulfite	EPA 377.1		P, G	500	Immed.
Surfactants	EPA 425.1		P, G/4°C	250	48 Hours
Total Organic Carbon	EPA 415.1	9060	P, G/4°C HCl pH < 2	100	28 Days
Total Organic Halogen	EPA 450.1	9020/9021	GA/4°C	500	14 Days
Turbidity	EPA 180.1		P, G/4°C	100	48 Hours

Sample Containers
P Plastic, polyethylene bottle with a polypropylene cap
G Glass
WK Whirl-Pak®
GA Glass, amber bottle with a Teflon® lined cap

Preservatives
H_2SO_4 Sulfuric Acid
HNO_3 Nitric Acid
NaOH Sodium Hydroxide

*Sample container, preferred volume and holding time are for water matrix. Consult laboratory for solid matrix sampling recommendations.

NOTE:
The methods shown are those commonly employed in performing environmental analyses. It is not intended to be inclusive of all possible EPA analytical methods or to indicate that any laboratory routinely provides the methods or parameters shown.

pace laboratories, inc.

Organic Analytical Guide

TABLE 1 (CONT.)

Water and Wastewater Analysis

EPA Method	Parameter	Technique	Sample Preparation	Sample Container/ Preservative	Preferred Volume (ml)	EPA Holding Time
601	Purgeable Halocarbons	GC-HALL	P&T	VOA/4°C	40	14 Days
602	Purgeable Aromatics	GC-PID	P&T	VOA/4°C	40	14 Days
603	Acrolein and Acrylonitrile	GC-FID	P&T	VOA/4°C, pH Adjusted	40	14 Days
604	Phenols	GC-FID	EXT	GA/4°C	1000	7/40 Days
605	Benzidines	HPLC-Electrochem	EXT	GA/4°C	1000	7/40 Days
606	Phthalate Esters	GC-ECD	EXT	GA/4°C	1000	7/40 Days
607	Nitrosamines	GC-NPD	EXT	GA/4°C	1000	7/40 Days
608	Organochlorine Pesticides and PCB's	GC-ECD	EXT	GA/4°C	1000	7/40 Days
609	Nitroaromatics and Isophorone	GC-FID + ECD	EXT	GA/4°C	1000	7/40 Days
610	Polynuclear Aromatic Hydrocarbons	HPLC-UV/Fluor or GC-FID	EXT	GA/4°C	1000	7/40 Days
611	Haloethers	GC-HALL	EXT	GA/4°C	1000	7/40 Days
612	Chlorinated Hydrocarbons	GC-ECD	EXT	GA/4°C	1000	7/40 Days
613	2, 3, 7, 8-Tetrachlorodibenzo-p-dioxin	GC/MS	EXT	GA/4°C	1000	7/40 Days
614	Organophosphonate Pesticides	GC-FPD or NPD	EXT	GA/4°C	1000	7/40 Days
615	Chlorinated Herbicides	GC-ECD or Hall	EXT	GA/4°C	1000	7/40 Days
624	Purgeables	GCMS	P&T	VOA/4°C	40	14 Days
625	Base/Neutrals, Acids and Pesticides	GC/MS	EXT	GA/4°C	1000	7/40 Days

Solid Waste Analysis

EPA Method	Parameter	Technique	Sample Preparation	Sample Container/ Preservative	Preferred Volume	EPA Holding Time
8010	Purgeables Halogenated Volatile Organics	GC-HALL	5030	VOA/4°C	*	14 Days
8015	Purgeables Non-Halogenated Volatile Organics	GC-FID	5030	VOA/4°C	*	14 Days
8020	Aromatic Volatile Organics	GC-PID	5030	VOA/4°C	*	14 Days
8030	Acrolein, Acrylonitrile, Acetonitrile	GC-FID	5030	VOA/4°C	*	14 Days
8040	Phenols	GC-FID	3550	GA/4°C	*	14 Days or 7/40 Days**
8060	Phthalate Esters	GC-ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8080	Organochlorine Pesticides and PCB's	GC-ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8090	Nitroaromatics and Cyclic Ketones	GC-FID or ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8100	Polynuclear Aromatic Hydrocarbons	GC-FID	3550	GA/4°C	*	14 Days or 7/40 Days**
8120	Chlorinated Hydrocarbons	GC-ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8140	Organophosphorus Pesticides	GC-FPD or NPD	3550	GA/4°C	*	14 Days or 7/40 Days**
8150	Chlorinated Herbicides	GC-ECD or HALL	3550	GA/4°C	*	14 Days or 7/40 Days**
8240	Volatile Organics	GC/MS	5030	VOA/4°C	*	14 Days
8250	Semi-Volatile Organics	GC/MS	3550	GA/4°C	*	14 Days or 7/40 Days**

Technique

Instruments:
- GC — Gas Chromatograph
- GC/MS — Gas Chromatograph-Mass Spectrometer
- HPLC — High Performance Liquid Chromatograph

Detectors:
- ECD — Electron Capture
- Fluor — Fluorescence
- FID — Flame Ionization
- FPD — Flame Photometric
- HALL — Electrolytic Conductivity
- NPD — Nitrogen Phosphorous
- PID — Photoionization
- UV — Ultraviolet

Sample Preparation Method Used:
- EXT — Extraction Methods that could be used include 3510, 3520, 3540 and 3550.
- P&T — Purge and Trap
- 3510 — Separatory Funnel Extraction of Liquid Samples
- 3520 — Continuous Liquid-Liquid Extraction
- 3540 — Soxhlet Extraction of Solid Samples
- 3550 — Sonication Extraction of Solid Samples
- 5030 — Purge and Trap, Direct Injection of Liquid Samples. Solid Samples Mixed then Injected.

Sample Container/Preferred Volume:
- GA — Glass Amber Bottle with Teflon Lined Cap
- VOA — Volatile Organic Analysis 40 ml Amber Glass Vial with Teflon Septum
- * — Contact Laboratory for recommendation

EPA/Holding Time:
- 7/40 — 7 Days for Extraction and 40 Days for Analysis
- ** — Depends upon Sample Matrix

NOTE:
The methods shown are those commonly employed in performing environmental analyses. It is not intended to be inclusive of all possible EPA analytical methods or to indicate that any laboratory routinely provides the methods or parameters shown.

TABLE 1 (CONT.)

Common Metals Analysis

	Solid	Water
Sample Container:	Plastic or glass	Plastic or glass
Preservative:	4°C	HNO_3 pH < 2
Preferred Amt.	100 grams	100 ml
EPA Holding Time:	6 Months	6 Months

Parameter	FLAME** EPA or Standard Method	FLAME** SW-846 Method	FURNACE EPA or Standard Method	FURNACE SW-846 Method
luminum	202.1	7020	202.2	NA
Antimony	204.1	7040	204.2	7041
Arsenic	206.3**	7060	206.2	7061
arium	208.1	7080	208.2	NA
erylium	210.1	7090	210.2	7091
Cadmium	213.1	7130	213.2	7131
alcium	215.1	7140	NA	NA
hromium, Total	218.1	7190	218.2	7191
Chromium, Hexavalent	Standard Method 312B	7195-7198	218.5	NA
obalt	219.1	7200	219.2	7201
opper	220.1	7210	220.2	NA
Gold	231.1	NA	231.2	NA
Iron, Total	236.1	7380	236.2	NA
ead	239.1	7420	239.2	7421
thium	Standard Method 317B	NA	NA	NA
Magnesium	242.1	7450	NA	NA
anganese	243.1	7460	243.2	NA
ercury (Cold Vapor)	245.1	7470/7471	NA	NA
Molybdenum	246.1	7480	246.2	7481
Nickel	249.1	7520	249.2	NA
otassium	258.1	7610	NA	NA
elenium	270.3**	7740	270.2	7741
Silicon	Standard Method 303C	NA	NA	NA
ver	272.1	7760	272.2	NA
odium	273.1	7770	NA	NA
Strontium	Standard Method 303A	NA	NA	NA
ellurium	Standard Method 303A	NA	Standard Method 304	NA
hallium	279.1	7840	279.2	7841
in	282.1	7870	282.2	NA
Titanium	283.1	NA	283.2	NA
anadium	286.1	7910	286.2	7911
nc	289.1	7950	289.2	NA

Metals by Inductively Coupled Plasma (ICP): Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Tl, V, Zn: EPA ICP Method 200.7 or SW-846 Method 6010

Flame A.A. or Hydride

B. SAMPLING PROCEDURE FOR GROUNDWATER AND SURFACE WATER

Groundwater and surface water sampling techniques employed by PACE are in accordance with the EPA Regional IV Standard Operating Procedures and Quality Assurance Manual, and the PACE Field Services SOP Manual.

Trained field sampling crews are dispatched to the site for sample collection and deliver collected samples to the laboratory.

For groundwater sampling, the water level within the well is determined prior to sampling using an electronic water level meter, then recorded on the field log data sheet with all additional pertinent information (Exhibit 4). The volume of water in the casing is calculated and three to five times that volume is purged from the well. In all cases, the well is purged until the conductivity, temperature, and pH have all stabilized, or the well has been purged dry.

Samples from monitoring wells are taken with a precleaned Teflon or stainless steel bailer. Bailers are precleaned by washing first with detergent, then rinsed with tap water, triple rinsed with deionized water, and baked at 105 C for one hour. Precleaned bailers are used between each sampling point.

All samples collected for metals analysis are preserved with nitric acid. The bailer to be used for sampling is used for purging two inch diameter wells and a gas-driven centrifugal pump is used when larger volumes of water need to be removed (static water levels of less than 25 feet). Wells with static water levels greater than 25 feet and casing diameters greater than 3 inches are purged using a submersible pump.

Quality Control Protocols:

A. All Quality Control (QC) procedures are as specifically required by the method, state, or project requirements.

B. The USEPA requires as a minimum one matrix spike, one duplicate or MSD, one blank, per set of samples of similar matrix with a maximum of 20 samples per set. This is a recommended minimum frequency for QC, unless stated otherwise by method, state or project requirements. A client may also request more frequent QC in which case it will be necessary to collect additional samples.

EXHIBIT 4

FIELD LOG DATA SHEET
PACE, Inc.
WELL SAMPLING

Client:_____ Project:_____ Project #:_____
Sample Site:_____
Well Identification and Description: (Locked_____ Not Locked_____) Key#:_____
ID inches_____PVC:_____Steel:_____Stainless Steel:_____Other:_____Labeled:_____
Total Well Depth (from top of casing)_____meters_____feet Elevation:_____feet
Static Water Level (from top of casing) Before Prepumping:_____meters_____feet
Static Water Level (from top of casing) At Time of Sampling:_____meters _____feet
Static Water Elevation:_____feet Water Column:_____feet One Casing Volume_____gal

Date Prepumped:_____Time Prepumped:_____Volume Prepumped:_____gal
Prepumping Method Used:_____ Pump Rate:_____gpm
Date Sampled:_____Time Sampled:_____Sampling Equipment Used:_____

Sample Temperature:_____°C Sample pH:_____Sample Specific Conductance:_____umho/cm2
Field Measurements Temperature Corrected: Yes___No___Metals Filtered in Field: Yes___No__
Weather Conditions:_____
Observations:_____
_____
Sample Description:_____
Name and Affiliation of Sampler(s)_____
Name and Affiliation of Inspector(s) Present:_____

STABILIZATION TEST

Time	pH	Specific Conductance (umhos/cm2)	Temp. (°C)	Cumulative Volume Removed (gallons)

C. SAMPLING PROCEDURES FOR SOILS AND SEDIMENTS

Soil and sediments are collected according to procedures in the latest edition of Test Methods for Evaluating Solid Waste, EPA-SW-846.

Soil sampling is designed to determine the depth and range of contamination from spillage or the leaching effects of rain on materials stored above ground. If borings are required, the depth and placement of the borings are planned by the project manager/subcontractor and client, using the suspected range of contamination as a guide.

VII. SAMPLE CUSTODY

A. SAMPLE RECEIPT

Sample shipments are received at the sample receiving area. Sample custodians verify the number of shipping containers received against the numbers listed on the shipping manifest/chain-of-custody. Any damage to the shipping containers or other discrepancy observed is noted on the chain-of-custody before signing it. A copy is kept for future reference.

The external chain-of-custody must be signed by the carrier for relinquishment of samples and signed by sample custodian personnel for sample receipt. The actual chain-of-custody may be supplied by PACE, (Exhibit 5), or may be the client's own form. The chain-of-custody remains in the project file at all times.

B. SAMPLE VERIFICATION

Upon arrival of a sample shipment, sample control personnel perform sample inspection. PACE's Sample I.D. and Condition Sheet (or equivalent) (Exhibit 6) serves as a check-off list of procedures to follow and as documentation of the following:

1. Presence/absence of custody seals or tapes of the shipping containers and the condition of the seals (i.e., intact, broken).

2. Presence/absence of chain-of-custody; (if present, is it complete?)

3. Presence/absence of sample tags; (if present, are they removable?)

4. Agreement/non-agreement between the sample tags, chain-of-custody, and any client documentation.

5. Condition of the samples when received, including:

   - Sample temperature
   - Intact, broken/leaking
   - Headspace in VOA vials
   - Sample holding time
   - Sample pH when required

   If discrepancies are found, the PACE project manager is contacted immediately (verbally and by using a Discrepancy Report Form) (Exhibit 7). If the project manager is not available, the QC manager is contacted for further directions. A copy of a Discrepancy Report Form is attached to the project data package.

pace INCORPORATED
THE ASSURANCE OF QUALITY

47955

EXHIBIT 5

CHAIN-OF-CUSTODY RECORD
Analytical Request

Client

Address

Phone

Report To:

Bill To:

P.O. # / Billing Reference

Project Name / No.

Pace Client No.

Pace Project Manager

Pace Project No.

*Requested Due Date:

Sampled By (PRINT):

Sampler Signature

Date Sampled

ITEM NO.	SAMPLE DESCRIPTION	TIME	MATRIX	PACE NO.	NO. OF CONTAINERS	PRESERVATIVES: UNPRESERVED	H_2SO_4	HNO_3	VOA			ANALYSES REQUEST	REMARKS
1													
2													
3													
4													
5													
6													
7													
8													

COOLER NOS.	BAILERS	SHIPMENT METHOD OUT / DATE	SHIPMENT METHOD RETURNED / DATE

ITEM NUMBER	RELINQUISHED BY / AFFILIATION	ACCEPTED BY / AFFILIATION	DATE	TIME

Additional Comments

29

ORIGINAL

SEE REVERSE SIDE FOR INSTRUCTIONS

SAMPLE I.D. AND CONDITION FORM

Client: _____
Project No.: _____
Date Received: _____

SAMPLE CONDITION UPON RECEIPT CHECKLIST

Complete checklist (A) during sample receipt. If any items are marked "NO," complete section (B) of this form. Otherwise, go to record samples.

			YES	NO
(A)	1.	Are there custody seals or tapes on the shipping container?	___	___
	2.	Are custody seals on the shipping container intact?	___	___
	3.	Is there a completed Chain-Of-Custody (C-O-C)?	___	___
	4.	Do the numbers of samples received and the sample matrices agree with C-O-C?	___	___
	5.	Are there tags attached to each sample?	___	___
	6.	Are sample tags, sample containers and C-O-C all in agreement?	___	___
	7.	Is the C-O-C complete with requested analyses?	___	___
	8.	Are the samples preserved correctly?	___	___
	9.	Is there enough sample to do all analyses?	___	___
	10.	Do the samples have the proper temperature?	___	___
	11.	Are the sample containers intact (e.g., not broken, leaking)?	___	___
	12.	Are VOA vials head-space free?	___	___
	13.	Are all samples within the holding times for requested analyses?	___	___
	14.	Is pH recorded for non-VOA's?	___	___

(B) Explain "NO" item here: _____

_____

_____

Send a copy of this form to Project Manager with Discrepancy Report Form. Copy of both forms remain in the QC file.

Custodian Signature: _____

EXHIBIT 7

PACE, INC.
DISCREPANCY REPORT FORM

Urgency Level: 1(_____) Requires immediate attention
2(_____) Requires attention today
3(_____) Requires attention this week

Initiator: _____ Client: _____
Date: _____
Project # _____

Sample(s) # _____ _____ _____ _____ _____

_____ _____ _____ _____ _____

_____ _____ _____ _____ _____

Discrepancy (if more space needed, use the back of this form): _____

_____

_____

To QC Manager: _____ Date: _____

Client Notified? YES ( ) NO ( ) Date & Time: _____

Project Manager Notified? YES ( ) NO ( ) Date & Time: _____

QC Response: _____

_____

_____

Project Manager Response: _____

_____

_____

Cause and Resolution (proposed or carried out): Completed by: _____

_____

_____

_____

Manager's Initials:

PM Signature: _____ Date: _____

QC Signature: _____ Date: _____

cc: Project File

C. SAMPLE LOG-IN

1. General Policies

a. Upon completing sample receipt/custody procedures, all sample and analysis data must be complete and documented on the chain-of-custody or accompanying forms for input into the Lab Data Management System (LDMS).

Sample and analysis data must include:

1. Client name and contact
2. Client number
3. PACE project number
4. PACE project manager
5. Sample descriptions
6. Due date
7. List of analyses requested

b. Sample and requested analyses data are input into the LDMS.

c. All samples received are logged into the LDMS on the day of receipt.

d. A Sample and Analysis Data Entry Form (SADEF) is generated immediately by the LDMS.

Distribution of SADEF:

- To the PACE Project Manager with a photocopy of the chain-of-custody. (Include a copy of the Discrepancy Report is applicable).
- To the QC project file with the original chain-of-custody.
- Photocopy to the Organic or Inorganic Department Manager as it applies for RUSH samples.
- To the client.

e. SADEF is to be reviewed against the chain-of-custody.

f. Sample containers are labeled with the corresponding sample number and the stamped date of receipt.

g. Samples are ready for storage.

2. **When Samples Are Received With No Paperwork**

a. If delivered by a client: Client is asked if previous arrangements were made for analysis (and with whom). The client completes a chain-of-custody and/or request for analysis, relinquishes samples to sample custodian personnel, and is given a copy of the C-O-C.

b. If received by courier or shipping:

1st: Routine Client File is checked
2nd: Anticipate Sample Alert File is checked
3rd: Sampling Kit Request File is checked
4th: PACE key client contact is consulted
5th: QC department manager is consulted to determine the designated PACE project manager
6th: Information is requested from the PACE project manager.

c. If analysis information can not be determined on the day of sample receipt, sample data entry personnel proceed to assign sample numbers and put samples on hold. Follow-up with project manager occurs until the analyses are determined and samples can be properly logged in.

3. **Responsibilities for Sample Log In**

a. Quality Control Manager/Sample Management Officer

- Has the overall responsibility for ensuring that this procedure is implemented for all samples received into the laboratory.
- Has overall responsibility for ensuring that samples are logged in correctly (given that appropriate information has been supplied).

b. Sample Custodian

- Has the primary responsibility of ensuring that sample information is input into the LDMS as described in the SOP.
- Has the responsibility to make recommendations to the QC manager for revising the SOP.

D. SAMPLE STORAGE

1. General Procedures

Samples for analysis are properly stored in the lab according to container type, preservative, and type of security required by the project.

Samples are stored immediately upon receipt to prevent sample degradation.

2. Refrigerated Storage Area Maintenance

All refrigerated storage areas are maintained at 1°- 4°C. The temperature is monitored and recorded daily. If the temperature fails outside the limit of 1°- 4°C, corrective action is to be taken as follows and appropriately documented.

a. Temperature is monitored at 30 minute intervals with the refrigerator door closed.

b. QC Manager is notified if the problem persists longer than one hour.

c. Samples are relocated to a proper storage environment if temperature cannot be maintained after corrective actions are implemented.

3. Routine Sample Storage

a. General Samples

Samples within each project are stored in sample number order. Waters and soils are generally stored on labeled separate shelves.

4. Specific Procedures

a. Volatiles

Samples within a project are stored in numerical order in vial containers. The holders are then stored where space permits in one of the designated volatile organic refrigerated storage areas.

b. Semi-Volatiles

Samples within a project are stored in numerical order in a designated, refrigerated storage area.

c. Hazardous Materials

Pure product or potentially heavily contaminated samples are tagged as "hazardous" and stored within a secured area, separate from other samples. This area is used only for hazardous samples and is labeled per OHSA requirements.

d. Special Projects

- Volatiles

  Samples within a project are stored in sample number order in vial containers. The holders are then stored as space permits in the Special Project VOA refrigerated storage area.

e. Asbestos

No refrigeration required. Samples are taken to asbestos lab for storage.

5. **Responsibilities for Sample Storage**

a. QC Department Manager/Sample Management Officer has direct responsibility for ensuring that the SOP is followed, samples are stored properly upon receipt, and refrigerated storage area temperatures are maintained.

b. Sample custodians are responsible for storing all samples upon receipt into the appropriate storage area, maintaining high level security for those samples under custody, and for keeping a current custody sample inventory.

c. Analytical personnel have the responsibility of daily sample storage area maintenance, disposal of old samples, and providing space for incoming samples in routine storage areas.

d. Assigned individuals are responsible for maintaining and documenting: (a) refrigerated storage area temperatures, and (b) corrective actions.

F. SAMPLE/DATA ACCESS AND INTERNAL CHAIN-OF-CUSTODY

1. General Policies and Procedures

PACE has implemented standard operating procedures to assure the integrity of samples and data so that they are not degraded or disclosed to unauthorized personnel. In order to ensure that this policy is maintained, the laboratory facilities are under controlled access. Only employees are allowed into the laboratory facilities; visitors must register at the front desk.

Samples are removed from their proper location by designated personnel and returned to the storage area immediately after the required sample quantity has been taken. This minimizes unnecessary time spent searching for samples and helps prevent matrix degradation from prolonged exposure to room temperature. After the final report is sent and clients are allowed adequate time to review the results, the samples are properly discarded or returned to the client.

PACE normally completes the sample analysis within 15 working days after receipt. Holding times may require faster turnaround times.

Upon client request, additional and more rigorous chain-of-custody protocols for samples and data can be implemented. For samples involving a high degree of confidentiality or potential litigation, PACE, Inc. has developed extensive sample and data handling protocols to assure the scientific and legal defensibility of the report submitted. These protocols include those specified by the USEPA Contract Laboratory Program.

Analysts and technicians follow strict internal chain-of-custody procedures to further ensure the validity of all data. All samples are signed out in a sample custody log book when they are removed for analysis. The sample ID, date, time, analyst, and lab of analysis is recorded in the sample custody log (Exhibit 8) or equivalent. Samples are signed back in noting date, time, and storage location, upon return.

EXHIBIT 8

SAMPLE CUSTODY LOG

Contract/Project No.: _____
Date Received: _____
Received by: _____
Time: _____
Witness: _____
Stored in: _____

Samples No(s). _____
_____
_____
_____
_____
_____

Date & Time Removed	Sample Nos. Removed	Name	Witness	Time Returned	Name	Witness

REMINDER: Samples must be returned at the end of the shift.

2. **Responsibilities for SOP Compliance**

a. The QC manager has the overall responsibility for ensuring that the SOP is implemented and followed.

b. The sample custodian personnel have the responsibility for ensuring that the SOP is properly followed, and to notify the QC manager of problems.

c. All employees checking out samples are required to follow procedures.

G. EXCESS SAMPLE DISPOSITION

Samples not totally consumed during the analyses are returned to the client. It is the project manager's responsibility to ensure that proper disposal has taken place. If the sample is water or wastewater and is considered non-hazardous by the project manager, it may then (by request) be properly disposed of at PACE facilities and not returned to the client.

1. **Notification of Sample Return**

The project manager and client receive written notification at the time of project initiation in the following manner:

a. The project proposal states the following paragraph in its Conditions and Terms Statement:

PACE, Inc. Standard Operating Procedures is to return all samples of hazardous materials or wastes to the client at project completion, and PACE, Inc. reserves the right to return or dispose of all samples at our discretion.

This is a standard form used by PACE's Marketing Department.

b. The Sample and Analysis Data Entry Form states the following sentence:

- PACE, Inc. reserves the right to return all samples at our discretion.

- This form is printed out by the LDMS at sample check-in.

c. The Sample and Analysis Data Entry Form cover letter will state the following paragraph:

1. PACE, Inc. Standard Operating Procedure is to return all samples of hazardous materials or wastes to the client at project completion. PACE, Inc. reserves the right to return or dispose of all samples at our discretion. (Exhibit 9) This is a pre-printed cover letter that accompanies the Sample and Analysis Data Entry Form.

d. The Sample and Analysis Data Entry Form and cover letter is sent to the project manager and to the client by the sample custodian personnel.

2. **Sample Return and Disposal**

Upon completion of laboratory analysis and/or the project, the LDMS automatically prints a report, invoice and sample disposition form. This form is part of the report package and is routed to the project manager.

a. The Sample Disposition Form (Exhibit 10) contains the following information:

1. Client name, address, and contact
2. PACE project number
3. Client project identification number
4. PACE sample identification number
5. PACE project manager name

pace
INCORPORATED
THE ASSURANCE OF QUALITY

EXHIBIT 9

August 29, 1991

Dear Valued Client:

A new policy has been implemented in the Sample Receiving Department of PACE, Inc. We hope that this policy will be helpful to you.

Upon receipt of samples into the laboratory, the Sample Custodian completes a Sample and Analysis Data Entry Form. This form is designed to accommodate a short description of the samples received (sample name and/or sample reference), the type of container, and a list of the analyses requested to be performed on each sample. A copy of this form will be sent to the Client (submitter).

Enclosed is a copy of the Sample and Analysis Data Entry Form relevant to the samples we recently received from you. Please compare the information on the form to assure that it is consistent with your request. If there is any inconsistency or if you have any questions on your project, please call the PACE Contact indicated on the Sample and Analysis Data Entry Form. The PACE Contact has primary responsibility for monitoring the progress of your project through the laboratory.

It is also part of PACE, Inc.'s Standard Operating Procedure to return all samples pertaining to the information attached that are hazardous materials or hazardous wastes to the client at project completion. PACE, Inc. reserves the right to return or dispose of all samples at our discretion.

We have implemented this procedure to better serve our clients, and would appreciate any comments you may have.

Sincerely,

1710 Douglas Drive North
Minneapolis, MN 55422
TEL: 612-544-5543
FAX: 612-525-3377

EXHIBIT 10

SAMPLE DISPOSITION FORM

_____

_____

_____

_____

_____

Date removed: _____
Initials: _____

Date shipped: _____
Initials: _____

RE: Client Project ID: _____

PACE Project No.: _____

Sample ID _____ _____ _____ _____

Dear _____:

All requested analyses of the samples for the above referenced project have been completed. Enclosed are the remaining portions of the samples which are being returned to you for final disposition.

If you have any questions, please call me.

Sincerely,

_____
Project Manager

3. Procedure for Use of the Sample Disposition Form

a. The project manager separates the sample disposition form from the report package, signs the form, and routes it to the sample custodian. If the sample is water or wastewater and non-hazardous, the project manager may wish to properly dispose of the waste.

- If the project requires, the project manager may hold the form for an acceptable amount of time before return or disposal.

- It is important that this form be used and not discarded. It is part of the internal chain-of-custody and is filed with the project report.

- The project manager will use action codes such as:

  1 = Return to client 2 = In house disposal
  C = Clean D = Dirty

  As a general rule, soil samples will be returned and water samples will be disposed of in-house. Water samples which are highly contaminated will be returned. Preserved samples, VOA's, and extracted/tainted samples will not be returned to the client. Therefore, it is necessary to note clean or dirty to facilitate handling. If a sample has an extremely high level of contamination, note the contaminant.

  For In-House Sample Disposal

  All preserved - Clean - Neutralize/sink
  Dirty - Toxic waste

  Un-preserved water - Clean - Sink
  Dirty - Toxic waste

  Soil/Sludge - Clean - Trash
  Dirty - Toxic waste

  All VOA's - Clean - Neutralize/sink
  Dirty - Toxic waste

All Extracted/Tainted Samples

CAM Extracts - Clean - Neutralize/sink
Dirty - Acid metals waste

Other Extracts - Toxic waste

Liquid/Unknown Misc. - Project manager specify

- Project manager will complete the sample disposition form and route it back to invoicing.
- The invoicing department will put completed sample disposition form in sample control mailbox.

b. Upon receipt of the Sample Disposition Form by the sample custodian personnel, the custodian personnel will remove the samples from storage using the information provided on the form.

- If the Sample Disposition Form indicates "Dump," the sample custodian personnel will remove them from storage and place them at a sample disposal station for proper disposal. The process of disposal is performed by the sample custodian personnel or appropriate laboratory staff. The Sample Disposition Form is signed and dated by the sample custodian personnel, then routed to the file clerk for filing with other project information.
- If the samples are to be returned, the sample custodian removes the sample or samples from storage, initials and dates the Sample Disposition Form. The samples, the Sample Disposition Form, and a copy of the client's chain-of-custody are then delivered to the shipping clerk by the sample custodian for return to the client.

c. Upon receipt of the samples and Sample Disposition Form, the shipping clerk signs and dates the form.

The Sample Disposition Form is copied and the original form with the samples is returned to the client, along with a copy of the client's chain-of-custody. A copy of the Sample Disposition Form and the original chain-of-custody is routed to the file clerk for filing with other project information (QC file).

- The shipping clerk labels the box with an appropriate hazard label and ships the samples back to the client using UPS or any other requested manner for shipment. (Note: It is important for proper packaging to prevent breakage during shipment.)

- All shipping costs will be charged against the appropriate project number.

d. Upon receipt of Sample Disposition Form, the file clerk files it with other project related information.

4. **Hazardous Material/Waste Sample Disposition Option**

The preferred method for disposition of excess hazardous material/waste samples is to return the excess sample to the client. It may not be feasible to return samples in all cases or the client may require PACE to dispose of excess samples. PACE will dispose of excess hazardous samples when required and will charge a disposal fee to recover costs for management and disposal.

Procedure for Disposal Option for Excess Hazardous Material/Waste Samples:

a. The project manager informs the client that excess sample disposal will require an additional charge.

b. When analyses are complete, the project manager indicates disposal as the option on the Sample Disposition Form and completes and attaches Hazardous Material/Waste Disposal Option Form (Exhibit 11). An entry is to be made in all fields of this form as it will determine the basis for lab packing and disposal.

c. The project manager routes the Disposal Option Form to sample check-in.

d. The project manager is responsible for billing the client for disposal.

e. The sample custodian is responsible for maintaining a file of Disposal Option Forms for all samples awaiting disposal. Hazardous material/waste samples are stored in safe manner, segregated by compatibility groups as indicated by the hazardous waste disposal SOP.

f. The Quality Control Manager is responsible for reviewing accumulated samples awaiting disposal and initiating the disposal process when warranted. The Field Services, Inorganic, Organic, and Environmental Services Departments cooperate and participate in the disposal process. (For compatibility and compositing, see the Hazardous Waste Disposal SOP.)

EXHIBIT 11

HAZARDOUS MATERIAL/WASTE SAMPLE
DISPOSAL OPTION FORM

Client _____ Client Project ID _____

Contact _____ PACE Project # _____

Address _____ Project Manager _____

_____ Report Sent Date _____

Phone Number _____ Pull Sample Date _____

Sample #	Matrix	Location	Disposal Method	Charge

Remarks: 1 = Return to Client
2 = In House Disposal
C = Clean
D = Dirty

Removed from Refrigerator (initial/date) _____

Returned to Client (initial/date) _____

Disposed of Samples (initial/date _____

VIII. CALIBRATION PROCEDURES AND FREQUENCY

Most measurements taken in the laboratory are based upon comparison to reference standards as analyzed by the standard method. The standard results are utilized to generate calibration curves or calibration factors. The results of the sample analysis are then quantified.

All instruments are calibrated using standard solutions of known concentrations. The standards are prepared from certified reference materials and are generally traceable back to NIST. Refer to Section XI for additional information.

The laboratory calibration procedures utilized meet or exceed the method calibration criteria for all analyses performed. If the method calibration requirements are more stringent than discussed in this document, the more stringent calibration requirements shall be achieved. The minimum instrument calibration procedures are discussed in Section IX by major instrument group; the calibration procedure in the method is followed for each specific analysis. Calibration procedures are documented on computer generated printouts and benchsheets where applicable.

Continuous calibration is verified by analysis of calibration standards or laboratory control samples from different sources at regular intervals. Recalibration is performed at specified time intervals or when indicated by the continuous verification procedure or as required by the method. Typical calibration and QC acceptance criteria for some common organic analyses are summarized in Table 2.

Forms to document initial and continuing calibration have been developed (Exhibits 12 and 13).

Refer to Section IX for additional calibration information and frequency as specified in the specific analytical methods.

TABLE 2 CALIBRATION AND QC ACCEPTANCE CRITERIA[a] FOR HALOGENATED VOLATILE ORGANICS

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Bromodichloromethane	15.2-24.8	4.3	10.7-32.0	42-172
Bromoform	14.7-25.3	4.7	5.0-29.3	13-159
Bromomethane	11.7-28.3	7.6	3.4-24.5	D-144
Carbon tetrachloride	13.7-26.3	5.6	11.8-25.3	43-143
Chlorobenzene	14.4-25.6	5.0	10.2-27.4	38-150
Chloroethane	15.4-24.6	4.4	11.3-25.2	46-137
2-Chloroethylvinyl ether	12.0-28.0	8.3	4.5-35.5	14-186
Chloroform	15.0-25.0	4.5	12.4-24.0	49-133
Chloromethane	11.9-28.1	7.4	D-34.9	D-193
Dibromochloromethane	13.1-26.9	6.3	7.9-35.1	24-191
1,2-Dichlorobenzene	14.0-26.0	5.5	1.7-38.9	D-208
1,3-Dichlorobenzene	9.9-30.1	9.1	6.2-32.6	7-187
1,4-Dichlorobenzene	13.9-26.1	5.5	11.5-25.5	42-143
1,1-Dichloroethane	16.8-23.2	3.2	11.2-24.6	47-132
1,2-Dichloroethane	14.3-25.7	5.2	13.0-26.5	51-147
1,1-Dichloroethene	12.6-27.4	6.6	10.2-27.3	28-167
trans-1,2-Dichloroethene	12.8-27.2	6.4	11.4-27.1	38-155
1,2-Dichloropropane	14.8-25.2	5.2	10.1-29.9	44-156
cis-1,3-Dichloropropene	12.8-27.2	7.3	6.2-33.8	22-178
trans-1,3-Dichloropropene	12.8-27.2	7.3	6.2-33.8	22-178
Methylene chloride	15.5-24.5	4.0	7.0-27.6	25-162
1,1,2,2-Tetrachloroethane	9.8-30.2	9.2	6.6-31.8	8-184
Tetrachloroethene	14.0-26.0	5.4	8.1-29.6	26-162
1,1,1-Trichloroethane	14.2-25.8	4.9	10.8-24.8	41-138
1,1,2-Trichloroethane	15.7-24.3	3.9	9.6-25.4	39-136
Trichloroethene	15.4-24.6	4.2	9.2-26.6	35-146
Trichlorofluoromethane	13.3-26.7	6.0	7.4-28.1	21-156
Vinyl chloride	13.7-26.3	5.7	8.2-29.9	28-163

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 601 and were calculated assuming a QC check sample concentration of 20 ug/L.

TABLE 2. CALIBRATION AND QC ACCEPTANCE CRITERIA[a] FOR AROMATIC VOLATILE ORGANICS

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Benzene	15.4-24.6	4.1	10.0-27.9	39-150
Chlorobenzene	16.1-23.9	3.5	12.7-25.4	55-135
1,2-Dichlorobenzene	13.6-26.4	5.8	10.6-27.6	37-154
1,3-Dichlorobenzene	14.5-25.5	5.0	12.8-25.5	50-141
1,4-Dichlorobenzene	13.9-26.1	5.5	11.6-25.5	42-143
Ethylbenzene	12.6-27.4	6.7	10.0-28.2	32-160
Toluene	15.5-24.5	4.0	11.2-27.7	46-148

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

[a]Criteria are from 40 CFR Part 136 for Method 602 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 1.

TABLE 2. QC ACCEPTANCE CRITERIA[a] FOR ORGANOCHLORINE PESTICIDES & PCB's

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Aldrin	2.0	0.42	1.08-2.24	42-122
α-BHC	2.0	0.48	.98-2.44	37-134
β-BHC	2.0	0.64	0.78-2.60	17-147
δ-BHC	2.0	0.72	1.01-2.37	19-140
γ-BHC	2.0	0.46	0.86-2.32	32-127
Chlordane	50	10.0	27.6-54.3	45-119
4,4'-DDD	10	2.8	4.8-12.6	31-141
4,4'-DDE	2.0	0.55	1.08-2.60	30-145
4,4'-DDT	10	3.6	4.6-13.7	25-160
Dieldrin	2.0	0.76	1.15-2.49	36-146
Endosulfan I	2.0	0.49	1.14-2.82	45-153
Endosulfan II	10	6.1	2.2-17.1	D-202
Endosulfan Sulfate	10	2.7	3.8-13.2	26-144
Endrin	10	3.7	5.1-12.6	30-147
Heptachlor	2.0	0.40	0.86-2.00	34-111
Heptachlor epoxide	2.0	0.41	1.13-2.63	37-142
Toxaphene	50	12.7	27.8-55.6	41-126
PCB-1016	50	10.0	30.5-51.5	50-114
PCB-1221	50	24.4	22.1-75.2	15-178
PCB-1232	50	17.9	14.0-98.5	10-215
PCB-1242	50	12.2	24.8-69.6	39-150
PCB-1248	50	15.9	29.0-70.2	38-158
PCB-1254	50	13.8	22.2-57.9	29-131
PCB-1260	50	10.4	18.7-54.9	8-127

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 2. CALIBRATION AND QC ACCEPTANCE CRITERIA[a] FOR GC/MS VOLATILE ORGANICS

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range p, p_s (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.4	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7	13.6-28.4	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8	1.0-39.0	D-227
trans-1,3-Dichloropropene	10.0-30.0	10.4	7.6-32.4	17-183
Ethyl benzene	11.8-28.2	7.5	17.4-26.7	37-162
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.5-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

Q = Concentration measured in QC check sample, in ug/L.
s = Standard deviation of four recovery measurements, in ug/L.
$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.
p, p_s = Percent recovery measured.
D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 624 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

TABLE 2. QC ACCEPTANCE CRITERIA[a] FOR GC/MS SEMIVOLATILE ORGANICS

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P_s, P_s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27.133
Benzo(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl)ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl)ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl)phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octylphthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113

TABLE 2. QC ACCEPTANCE CRITERIA[a] FOR GC/MS SEMIVOLATILE ORGANICS (CONT.)

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range p, p_s (%)
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

p, p_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 625. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

INITIAL CALIBRATION DATA
EXTRACTABLE 8080/608 COMPOUNDS
EXHIBIT 12

CALIBRATION DATE: COLUMN ID:

INSTRUMENT ID: DETECTOR ID:

MAXIMUM % RSD IS 20%

Standard ID	#	#	#		
Compound	CF 20	CF 40	CF 60	$\overline{CF}$	%RSD
Alpha-BHC					
Beta- BHC					
Lindane					
Delta- BHC					
Heptachlor					
Aldrin					
Heptachlor Epoxide					
Endosulfan I					
DDE/Dieldrin					
Endrin					
Endosulfan II					
4,4'-DDD					
Endrin Aldehyde					
4,4'-DDT					
Endosulfan Sylfate					

CF=CALIBRATION FACTOR= $\frac{\text{Total ng of Standard}}{\text{Area}}$

$\overline{CF}$ = AVERAGE CALIBRATION FACTOR = CF/n

%RSD = RELATIVE STANDARD DEVIATION = $\frac{\text{(Standard Dev.) (100)}}{\overline{CF}}$

EXHIBIT 13

CONTINUING CALIBRATION CHECK
Semi-Volatile Compounds

CASE NO:_____ CALIBRATION DATE:_____

LABORATORY NAME: PACE LABORATORIES TIME:_____

CONTRACT/PROJECT NO._____ ANALYST:_____

INSTRUMENT I.D.:_____ INITIAL CALIBRATION DATE:___

MAXIMUM %D FOR CCC IS 10%

COMPOUND	CF	CF	%D	CCC
Alpha-BHC				
* Beta-BHC				
Lindane				
Delta-BHC				
Heptachlor				
* Aldrin				
Heptachlor Epoxide				
* Endosulfan I				
DDF/Dieldrin				
* Endrinion				
Endosulfan II				
4,4'-DDD				
Endrin Aldehyde				
4,4'-DDT				
Endosulfan Sulfate				
Aroclor 1016				
Aroclor 1221				
Aroclor 1232				
Aroclor 1242				
Aroclor 1248				
Aroclor 1254				
Aroclor 1260e				
Chlordane				
Toxaphene				
Methoxychor				
DBC				

CF -Calibration Factor from daily standard at ug/L
CF-Average Calibration Factor from initial calibration Form VI
%D-Percent Difference
CCC-Calibration Check Compounds

IX. ANALYTICAL PROCEDURES

Analytical methods used at PACE are EPA methodologies, where available, such as those specified in References 2 and 3 or approved equivalent methods. A list of typical analytical methods utilized at PACE is as follows:

A. LIST OF ANALYTICAL METHODS

1. Organic Analyses

Parameter	Method	DW	WW Method	SW 846	Spec.
Purgeable Halocarbons	GC	502.1/502.2	601	8010	
Non-Halogenated Volatile Organics	GC			8015	
Purgeable Aromatics and Unsaturated Organics	GC	503.1/502.2	602	8020	
Acrolein & Acrylonitrile	GC		603	8030	
Phenols	GC	515.1	604	8040	
Benzidines	HPLC		605		
Phthalate Esters	GC		606	8060	
Nitrosamines	GC		607		
Organochlorine Pesticides and PCBs	GC	508/505/508A 507/515.1	608/608.1 608.2	8080	(CA)Mod 8080 (MN)570A
Nitroaromatics and Isophorone	GC		609	8090	
Polynuclear Aromatic Hydrocarbons	HPLC/GC		610	8310/ 8100	
Haloethers	GC		611		
Alachlor, Atrazine, Chlordane, Hepatchlor, Heptachlor Epoxide, Lindane, Methoxychlor, Toxaphene, and PCBs (as Aroclors)	GC	505/507	645		MN 570A

Parameter	Method	DW	WW Method	SW 846	Spec.
Chlorinated Hydrocarbons	GC		612	8120	
2, 3, 7, 8 - TCDD	GC/MS		613		
Volatile Organics					(MN)465C
Base/Neutrals & Acids	GC/MS	525 (NCA)	625	8250/8270	
Organophosphorus Pesticides	GC	507	614/622	8140/ 8220	(MN)570A
Chlorinated Herbicides	GC	515.1	615/608.1/ 608.2	8150	(MN)574A (CA)509B
EDB and DBCP	GC	504			(CA) DOHS/ 8011
Volatile Organic Compounds	GC/MS	524.2/524.1	624	8240	8260
Carbamates & Urea & Pesticides	HPLC	531.1	632		(MN)572A (MN)572A
Fuel Hydrocarbons & BTEX	GC or IR		602/418.1	8020	(CA)9073 Mod. 8015
Alachlor, Atrazine	GC	507/505	619/645		(MN)570A (MN)570A
Chlordane, Heptachlor, Heptachlor Expoxide, Lindane; Methoxychlor	GC	508/505	608/617	8080	8081
Aldicarb; Aldicarb sulfone; Aldicarb sulfoxide; Carbofuran	GC	531.1			

2. Inorganic Analyses

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	ASTM	SW 846
A. Non Metals					
Acidity	Potentiometric Titration	402	305.1	D1067	
Alkalinity	Potentiometric Titration	403	310.1	D1067	
Bacteria,					
Total Coliform	Membrane Filter	909A			9132/9131
Fecal Coliform	Membrane Filter	908C			
Fecal Strept.	Membrane Filter	910A			
Total Plate Count	Agar Medium	907			
Biochemical Oxygen Demand, 5-Day	Winkler Electrode	507 507	405.1		
Boron	Curcumin 405-A	404A	212.3		
	ICP		200.7		6010
Chemical Oxygen Demand	Dichromate Reflux (High)	508A	410.1	D1252	
	Dichromate Reflux (Low)	508A	410.2	D1252	
Chloride	Mercuric Nitrate	407B	325.3	D512	9252
	Auto. Ferricyanide	407D	325.2		9251
	Titration	407A			
Chlorine, Residual	Amperometric Titration	408C	330.1 330.5	D1253	
	Colorimetric	408E			
Color	Visual Comparison	204A	110.2		
Cyanide, Total	Pyridine-Barbitutic Acid, Colorimetric	412D	335.2	D2036	9010A
Amenable	Chlorination-Colorimetric	412F	335.1	D2036	9010A 9012
Flouride, Total	Distillation-Electrode	413A/B	340.2	D1179	
Flouride, Diss.	Electrode	413B	340.2	D1179	

Parameter	Method	Standard Methods 15th Ed.	EPA Methods	ASTM	SW 846
Hardness, Total	EDTA Titration Calculation	314B	130.2	D1126	
Hardness, Calcium	EDTA Titration	303A	242.1	D511	
Ion Chromatography			300.0		9056
Nitrogen,					
Ammonia	Distillation Titration	417D	350.2		
	Potentiometric		350.3		
Kjeldahl	Digestion Distillation	420B	351.3	D3590	
Nitrate	Automated Cadmium	418F	353.2	D3867	9200
	Brucine Sulfate		352.1	D091	
Nitrite	Automated Cadmium	418F	353.2	D3867	
	Colorimetric	419			
Organic	Kjeldahl-NH_3	420A	351.3	D3590	
	Kjeldahl-Potentiometric		351.4		
Oil & Grease	Soxhlet	503C			
	Partition-Gravimetric	503A	413.1		9070/
			413.2		9071
Oxygen	Winkler	421B	360.2	D888	
Dissolved	Electrode	421F	360.1		
pH (Hydrogen Ion)	Electrode	423	150.1	D1293	9040
					9045
Phenol	Distillation-Extraction Colorimetric		420.1	D1783	9066
					9065
					9069
Phosphorus,	Persulfate Digestion-	424C/F	365.2	D515	
Total	Ascorbic Acid Reduc.				
Ortho	Ascorbic Acid Reduc.	424F	365.2	D515	
Silica,	Molybdosilicate	425C	370.1	D859	
Dissolved	ICP		200.7		
Solids					
Total	Gravimetric	209A	160.3		
Total Volatile	Gravimetric	209D	160.4		
Suspended	Gravimetric	209C	160.2		
Suspended Volatile	Gravimetric	209D	160.4		
Total Dissolved	Gravimetric	209B	160.1		
Settleable	Gravimetric	209E	160.5		
TOC			415.1		9060
TOX					9022

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	ASTM	SW 846
Specific Conductance	Meter	205	120.1	D1125	9040
Sulfate	Ion Chromatography	426C	300.0	D516	9035
	Automated Methyl Thymol Blue		375.2		9038
	Turbidimetric		375.4		9036
Sulfide	Colorimetric	427C	376.2		9030
	Titration	427D	376.1		
Sulfite	Titration	428A	377.1	D1339	
Surfactants (MBAS)	Methylene Blue	512B	425.1	D2330	
Turbidity	Meter	214A	180.1	D1889	

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1979	SW 846
B. Metals				
Aluminum	AA-Direct Aspiration	303C	202.1	7020
	AA-Furnace	304	202.2	
	ICP-AES		200.7	6010
Antimony	AA-Direct Aspiration	303A	204.1	7040
	AA-Furnace	304	204.2	7041
	ICP-AES		200.7	6010
Arsenic	AA-Gaseous Hydride	303E	206.3	7061
	AA-Furnace	304	206.2	7060
	ICP-AES		200.7	6010

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	SW 846
Barium	AA-Direct Aspiration	303C	208.1	7080
	AA-Furnace	304	208.2	7081
	ICP-AES		200.7	6010
Beryllium	AA-Direct Aspiration	303C	210.1	7090
	AA-Furnace	304	210.2	7091
	ICP-AES		200.7	6010
Cadmium	AA-Direct Aspiration	303A	213.1	7130
	AA-Furnace	304	213.2	7131
	ICP-AES		200.7	6010
Calcium	AA-Direct Aspiration	303A	215.1	7140
	AA-Furnace	311C	215.2	
	ICP-AES		200.7	6010
Chromium, Total	AA-Direct Aspiration	303A	218.1	7190
Hexavalent	AA-Furance	304	218.2	7191
	ICP AES		200.7	6010
	Colorimetric	312B		7196
	MIBK Extraction			7197
Cobalt	AA-Direct Aspiration	303A	219.1	7200
	AA-Furnace	304	219.2	7201
	ICP-AES		200.7	6010
Copper	AA-Direct Aspiration	303A	220.1	7210
	AA-Furnace	304	220.2	7211
	ICP-AES		200.7	6010
Iron	AA-Direct Aspiration	303B	236.1	7380
	AA-Furnace	304	236.2	7381
	ICP-AES		200.7	6010
Lead	AA-Direct Aspiration	303A	239.1	7240
	AA-Furnace	304	239.2	7241
	ICP-AES		200.7	6010

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	SW 846
Lithium	AA-Direct Aspiration	317B		
Magnesium	AA-Direct Aspiration	303A	242.1	7450
	ICP AES		200.7	6010
Manganese	AA-Direct Aspiration	303A	243.1	7460
	AA-Furnace	304	243.2	7461
	ICP AES		200.7	6010
Mercury	AA-Cold Vapor	303F	245.1	7470 or 7471
Molybdenum	AA-Direct Aspiration	303C	246.1	7480
	AA-Furnace	304	246.2	7481
Nickel	AA-Direct Aspiration	303A	249.1	7520
	AA-Furnace	304	249.2	
	ICP AES		200.7	6010
Potassium	AA-Direct Aspiration	303A	258.1	7610
Selenium	AA-Gaseous Hydride	303E	270.3	7740
	AA-Furnace	304	270.2	7741
	ICP AES		200.7	6010
Silver	AA-Direct Aspiration	303A	272.1	7760
	AA-Furnace	304	272.2	7761
	ICP AES		200.7	6010
Sodium	AA-Direct Aspiration	303A	273.1	7770
	ICP AES		200.7	6010
Strontium	AA-Direct Aspiration	303A		7780
Thallium	AA-Direct Aspiration	303A	279.1	7840
	AA-Furnace	304	279.2	7841
	ICP AES		200.7	6010
Tin	AA-Direct Aspiration	303A	282.1	7870
	AA-Furnace	304	282.2	

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	SW 846
Titanium	AA-Direct Aspiration	303C	283.1	
	AA-Furnace	304	283.2	
Vanadium	AA-Direct Aspiration	303C	286.1	7910
	AA-Furnace	304	286.2	7911
	ICP AES		200.7	6010
Zinc	AA-Direct Aspiration	303A	289.1	7950
	AA-Furnace	304	289.2	7951
	ICP AES		200.7	6010

3. **Wastes & Oil Analysis**

Parameter	Method	Standard Methods 15th Ed.	ASTM	SW 846
% Ash	Gravimetric	209F		
% Chlorine	Bomb Calorimeter		D808-81	
Density	Gravimetric	213E		
Flash Point Closed Cup	Tag		D93-80	1010
Free Liquids	Paint Filter			9095
Heat of Combustion	Bomb Calorimeter		D240-76	
Leach Test. EP Toxicity	Extraction			1310
ASTM Water	Extraction		D3987-85	
% Sulfur	Bomb Calorimeter		D129-64	

Parameter	Method	Standard Methods 15th Ed.	ASTM
Viscosity	Saybolt		D88-81
% Water	Distillation		D95-83

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1982	SW 846
Sulfide, Total	Titration			9030
Reactive	Titration		261.23	Chap. 7 7.3.4.2
pH	Electrode			9040
Specific Conductance	Meter			9050 9045
Specific Gravity	Mass Displacement	213E		
Cyanide, Total	Pyridine-Barbitric Acid Colorimetic			9010
Amenable	Chlorination-Colorimetric			9010
Cyanide, Reactive	Pyridine-Barbitric Acid Colorimetric		261.23	Chap. 7 7.3.3.2
TCLP			40CFR268	1311

4. Industrial Hygiene

A. Organic

Parameter	Method	NIOSH	OSHA
Acetic acid	GC-FID	1603	
Acrolein	GC-NPD		52
Acrylonitrile	GC-FID	1604	
Alcohols I	GC-FID	1400	
Alcohols II	GC-FID	1401	
Alcohols III	GC-FID	1402	
Alcohols IV	GC-FID	1403	
Amines,Aliphatic	GC-FID	221	
Aminoethanol	GC-FID	2007	
1,3-Butadiene	GC-FID	1024	
2-butanone (MEK)	GC-FID	2500	
n-butyl glycidyl ether	GC-FID	S-81	7
Dibutyl phthalate	GC-FID	5020	
Dichlorodifluoromethane	GC-FID	1018	
Dimethylacetmide	GC-FID	2004	
Dimethylforamide	GC-FID	2004	
Dioxane	GC-FID	1602	
Dipropylene glycol methyl ether	GC-FID	S-69	7
Endrin	GC-ECD	5519	
Epichlorohydrin	GC-FID	1010	
Estets I	GC-FID	1450	
Ethyl acetate	GC-FID	S-49	7
Ethylene dibromide	GC-FID	1008	
Ethylene glycol	GC-FID	5500	
Ethylene oxide	GC-ECD	1614	50
Ethyl ether	GC-FID	1610	
Fluorotrichloromethane	GC-FID	S-102	
Formaldehyde	GC-NPD		52
Glutaraldehyde	GC-FID	2531	
Glutaraldehyde	HPLC		64
Hydrazine	GC-FID/NPD	248	
Hydrocarbons	GC-FID	1500	7
Hydrocarbons,Aromatic	GGC-FID	1501	7
Hydrocarbons,Halogenated	GC-FID	1003	7
Isocyanates	HPLC-UV		42:47
Isophorone	GC-FID	2508	
Ketones I	GC-FID	1300	
Ketones II	GC-FID	1301	
Methanol	GC-FID	2000	
Methyl Cellosolve Acetate	GC-FID	S-39	
Methylene Chloride	GC-FID	1005	
Methyl Methacrylate	GC-FID	2537	
Mineral Oil mist	IR	5026	
Morpholine	GC	S-150	
Naphthas	GC-FID	1550	

Parameter	Method	NIOSH	OSHA
Nicotine	GC	S-293	
Pentachlorophenol	HPLC-UV	5512	39
Phenol	HPLC		32
Phenyl Glycidyl Ether	GC-FID	S-74	
Polynuclear Aromatic	HPLC-UV	5506	
Polynuclear Hydrocarbons	GC-FID	5515	
Propylene glycol monomethyl ether	GC-FID		53
1,1,1,2-Tetrachloro-2,2-difluoroethane	GC-FID	1016	
1,1,2,2-Tetrachloro-1,2-difluoroethane	GC-FID	1016	
1,1,2,2-tetrachloroethane	GC-FID	1019	
Tetrahydrofuran	GC-FID	1609	
Trichloroethylene (TCE)	GC-FID	1022	
1,1,2-trichloro-1,2,2-trifluoroethane	GC-FID	1020	
Vinyl acetate	GC-FID	278	51
B. Metals			
Aluminum	AA, ICP	7013,7300	
Arsenic	AA, ICP		ID-105, 7300
Arsenic trioxide	GFAA	7901	
Arsine	GFAA	6001	
Barium	AA	7056	ID-121
Beryllium	GFAA, ICP	7102,7300	
Cadmium	AA, ICP	7048,7300	
Calcium	AA, ICP	7020,7300	ID-121
Chromium	AA, ICP	7024,7300	ID-121
Chromium, Hexavalent	UV-VIS	7600,S-317	
Cobalt	AA, ICP	7027,7300	
Copper	AA, ICP	7029,7300	
Indium	AA, ICP	173,7300	ID-121
Iron oxide	AA, ICP	173,7300	
Lead	AA, ICP	7082,7300	
Lithium	AA, ICP	173,7300	
Magnesium	AA, ICP	173,7300	
Manganese	AA, ICP	173,7300	ID-121
Molybdemium	AA, ICP	173,7300	ID-121
Nickel	AA, ICP	173,7300	ID-121
Palladium	AA	173	ID-121
Potassium	AA	173	ID-121
Selenium	AA		ID-105
Silver	AA, ICP	173,7300	ID-121
Sodium	AA, ICP	173,7300	
Strontium	AA	173	
Tellurium	AA, ICP	173,7300	ID-121
Thallium	AA, ICP	173,7300	
Tin - inorganic	AA, ICP	S-183,7300	ID-121
Titanium	AA, ICP	7300	ID-121
Tungsten	AA, ICP	7074,7300	
Vanadium	AA, ICP	173,7300	

Parameter	Method	NIOSH	OSHA
Yttrium	AA, ICP	7300	ID-121
Zinc	AA, ICP	7030, 7300	ID-121
C. Inorganic			
Acids, inorganic	IC	7903	
Ammonia	colorimetric	205	
	IC	6701	
Asbestos, bulk	PLM	9002	
Asbestos, fiber	TEM	7402	
Carbon black	gravimetric	5000	
Chlorine	colorimetric	209	ID-101
Cyanides, aerosol and gas	ISE	7904	
Fibers (including asbestos)	PCM	7400	
Fluorides, aerosol and gas	ISE	7902	
Hydrogen Peroxide	UV-VIS		V1-6
Hydrogen sulfide	UV-VIS	S-4	
Iodine	IC	6005	
Nitrogen dioxide	UV-VIS	6700,231	
Nuisance dust, respirable	gravimetric	0600	
Nuisance dust, total	gravimetric	0500	
Ozone	UV-VIS	S-8	
Silica	XRD	7500	
Stibine	colorimetric	6008	
Sulfuric acid	titration	S-174	
Sulfur dioxide	titration	163	
Wood dust	gravimetric	0500	

4. **List of Sample Preparation Methods**

1311	TCLP
1312	Synthetic precipitation leaching procedure
3015	Microwave dig. aqueous
3051	Microwave dig. sludges, oil soil
3510	Separatory Funnel Liquid - Extraction
3520	Continuous Liquid - Extraction
3540	Soxhlet Extraction
3541	Automatic soxhlet extraction
3550	Sonication Extraction
3640	Gel Permeation Chromatography
3580	Waste Dilution
3630	Silica gel
3660	Sulfur clean up
5050	Bomb combus. method for T. Hal
5080	Purge and Trap
3005	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by Flame AA or ICP
3010	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame AA or ICP
3020	Acid Digestion of Aqueous Samples and Extracts for Total Metals by Furnace AA
3050	Acid Digestion of Sediments, Soils, and Sludges

Method numbers refer to EPA Methods except:

5. Screening Methods

3810	Headspace
3820	Hexadecane extraction and screening of purgeable organics

1. S.M. = Standard Methods for the Examination of Water and Wastewater
2. USATHAMA = U.S. Army Toxic and Hazardous Materials Agency
3. NIOSH = Manual of Analytical Methods
4. Hach, Chevron, Calgon = Industrial Methods

6. **List of Radiochemistry Methods**

900.0	Gross Alpha/Beta
903.1	Radium-226
904.1	Radium-228
708	Tritium
901.1	Gamma Scan
704	Strontium-89/90
908.0	Total Uranium

List of radiochemistry Methods (Cont.)

PACE	Americium, Isotopic
PACE	Plutonium, Isotopic
PACE	Thorium, Isotopic
PACE	Uranium, Isotopic
624/8240	Purgeable Volatiles ((GC/MS)
625/8270	BNA Extractable Semivolatiles (GC/MS)
608/8080	Organochlorine Pesticides and PCBs (GC/ECD)
615/8150	Phenoxy-Acid Herbicides (GC/ECD)
40 CFR 261	Characteristic of Ignitability
40 CFR 261	Characteristic of Corrosivity
40 CFR 261	Characteristic of Reactivity
40 CFR 261	TCLP
NIOSH 7400-A	Airborne Fibers (PCM)
600/M4-82-020	Bulk Asbestos (PLM)
NIOSH 0600	Nuisance Dust, Respirable
NIOSH 0500	Nuisance Dust, Total
NIOSH 7500	Respirable Silica (XRD)

B. GAS CHROMATOGRAPHY PROCEDURES

1. Calibration and Calibration Verification

All GC methods are calibrated by external calibration procedures using three to five standard concentrations, depending upon the method. A new calibration is performed at least once per quarter or as needed on routine analyses. Methods not utilized on a daily basis are calibrated before each run.

2. Laboratory Control Sample (LCS)

An NIST traceable external check sample is analyzed at least once per week, and when a new initial calibration is performed.

3. Matrix Spike

Performed at a minimum of every 20 samples or as required by either state or project-specific requirements.

4. Surrogate Spike

Surrogates are added to and analyzed for in every sample per applicable organic methodologies.

5. Duplicate Sample Analysis

Performed at a minimum of every 20 samples or as specified by state/project requirements. The matrix spike is duplicated.

6. Blank Analysis

The reagent/method blank must have no contaminants greater than the detection limit of the method. In the case of volatile organic analysis, common laboratory solvents may be present at a concentration of less than 5 times the MDL.

7. Surrogate Spikes - Surrogate spiking compounds are added to and analyzed for, with every sample including blanks. A surrogate is a compound chemically similar to a targeted analyte which is added to samples prior to purging or extraction.

8. Other

Method 608/8080 are also subject to the following QC criteria:

a. Combined breakdown of endrin and DDT may not exceed 20%. This is monitored through the daily analysis of an LCS containing these compounds.

b. Two LCS (each containing 1/2 the compounds of the method) are alternately analyzed after every tenth sample.

C. GAS CHROMATOGRAPHY/MASS SPECTROMETRY PROCEDURES

1. Calibration and Continuing Calibration

A minimum of an internal three point calibration is performed when indicated by the continuing calibration. One check standard is analyzed at the beginning of each 12-hour shift to verify calibration. The acceptance limit for the check standard is 25% RSD. Recalibration is necessary from once per week to once per month. Fresh calibration standards must be prepared weekly or as needed.

2. Validation of Mass Spectrometer

The mass spectrometers are tuned at the start of each run period and at 12-hour intervals. The tuning procedure utilizes the EPA/SW-846 recommended compounds 4-bromofluorobenzene (BFB) for 624/8240, 8260 and decafluorotriphenyl phosphine (DFTPP) for 625/8270.

3. Internal Standards

All sample results are quantified using the internal standard technique described in EPA methods 624, 8240, 8260, 625, 8270. Three (VOA) or six (BNA) internal standard compounds are added to each sample immediately before analysis. The internal standard nearest the retention time of the analyte of interest is used in the quantitation of the analyte.

4. Laboratory Control Sample

An EPA check sample (or external standard if EPA is unavailable) is analyzed at a minimum once every month. A standard is run every 12 hour shift.

5. Matrix Spike and Matrix Spike Duplicate

Performed at a minimum of every 20 samples or as specified by state/project requirements. This is the same procedure as the GC section.

6. Surrogate Spikes

Surrogate spiking compounds are added to and analyzed for, with every sample including blanks. A surrrogate is a compound chemically similar to a targeted analyte which is added to samples prior to purging or extraction.

7. **Reagent/Method Blank**

VOA - one per 12-hour per shift
BNA - one per batch of samples extracted

Common laboratory solvents present in the blank at a concentration less than 3 times the MDL will be footnoted on the analysis report. Common solvents at greater concentrations or the presence of any contaminant not considered a common laboratory solvent at a concentration greater than the MDL indicates the need to re-extract/re-analyze the blank and associated samples.

D. **METALS PROCEDURES**

1. **Calibration and Calibration Verification**

All instruments are calibrated at the start of each run. The graphite furnace requires 4 point calibration. The Flame AA and ICP methods utilize a minimum of 3 points. Cold vapor analysis of mercury requires a 5 point calibration. Recalibration is performed after 20 samples, or more often if indicated by the laboratory control sample. ICP uses calibration procedures as stated in the method.

2. **Laboratory Control Sample**

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

3. **Matrix Spike**

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

4. **Duplicate Samples**

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

5. **Blank Analysis**

a. Method Blank

If the concentration of the blank exceeds the MDL, all samples associated with the blank are redigested and reanalyzed concurrent with a new blank.

b. Reagent Blank

Any reagent blank result greater than the MDL terminates the analysis until corrective action resolves the problem. For ICP metals, a negative blank value greater than two times the MDL also requires corrective action. In rare cases, if all corrective action fails to resolve the problem, sample and blank data are reported if the problem cannot be rectified.

E. GENERAL CHEMISTRY PROCEDURES

1. Calibration and Verification

All instruments are calibrated daily with 3-6 point curves, depending upon instrument requirements. The calibration is continuously verified throughout the run, with either a calibration standard or laboratory control standard inserted after every 10th sample.

2. Laboratory Control Sample

A laboratory control sample is analyzed at least once during each batch of samples.

3. Matrix Spike and Duplicate Samples

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

F. RECORD KEEPING AND REVIEW

All records and data are generally logged into hard cover bound books.

The extractions section utilizes method-specific bound books to record all data pertaining to sample extraction and preparation. An extraction benchsheet is used to transfer information to GC or GC/MS with each extracted sample or batch (Exhibit 14 and 15).

The organic and inorganic departments utilize benchsheets, maintained by analysts; specific for injection data and instrument maintenance. Spectras and chromatograms are filed by acquisition date.

The individual analysts and technicians are responsible for maintaining accurate, legible records and logs in accordance with standard operating procedures. The supervisors are responsible for ensuring adherence to procedures.

Secondary review of all records and logs is performed by someone other than the person generating the document, preferably the department supervisor. Evidence of secondary review is provided on the document as initials and review date by the secondary person.

See Section X for magnetic media storage.

Exhibit 14

PROJECT #

GC-MS EXTRACTABLES

BATCH #

Sample Location	Sample Number	Date/Time of Extraction	Initial Volume	Surrogate	Spike	Final Volume	Date of Conc.	% Emulsion	Comments	Extract Location

EXTRACTION METHOD

- Separatory Funnel ☐
- Continuous Liq/Liq ☐
- Soxhlet ☐
- Sonication ☐
- Other: _____ ☐

Spike #

Dup. Spike #

QUALITY CONTROL INFORMATION

Surrogate:

Spike:

ROUTING

Person Who:	Initial
Extracted	_____
Concentrated	_____
Supervisor	_____
GC/MS	_____

WPPWGJ5

Exhibit 15

PROJECT #

GC EXTRACTION

BATCH #

Location	Sample Number	Weight of Sample	Date & Time of Extraction	Final Volume	Date of Conc.	% Emulsion	Comments	Extraction Location	Column	NCLSON File	% Recovery	Date & Time

EXTRACTION METHOD

Separatory Funnel ☐

Continuous Liq/Liq ☐

Soxhlet ☐

Sonication ☐

Other: _____ ☐

Spike #

Dup. Spike #

QUALITY CONTROL INFORMATION

Surrogate:

Spike:

ROUTING

Person Who:	Initial
Extracted	_____
Concentrated	_____
Supervisor	_____
GC/MS	_____

G. ACCEPTANCE CRITERIA AND CONTROL CHARTS

General acceptance criteria for quality control samples and instrument calibration/verification are summarized in Table 3. Internal in-house control limits are regionally generated for specific methodologies and instruments to achieve methodology and regulatory requirements.

TABLE #3

ACCEPTANCE CRITERIA FOR QUALITY CONTROL SAMPLES & INSTRUMENT CALIBRATION

	MATRIX SPIKE % RECOVERY	SURROGATE SPIKE % RECOVERY	RPD DUPLICATE SAMPLES	INITIAL CALIBRATION LINEARITY	CALIBRATION VERIFICATION	LCS/EPA QC SAMPLE
GC	Within calculated control limits*	Within calculated control limits	≤ maximum RPD acceptance limit	RSD ≤ 20%	± 15% of true value or initial response	±15% of true value or EPA limit
MS	Within calculated control limits*	Within calculated control limits	≤ maximum RPD acceptance limit	RSD ≤ 30%	± 30% of initial average RF	± 15% of true value or EPA limit
GENERAL CHEMISTRY	Within calculated control limits*	N/A	0-67 on samples < 10x MDL 0-20 on samples > 10x MDL MDL = Method Detection Limit	Correlation coefficient ≥ .995	± 10% of true value	± 15% of true value or EPA limit
METALS	Within calculated control limits*	N/A	0-67 on samples < 10x MDL 0-20 on samples > 10x MDL MDL = Method Detection Limit	Correlation coefficient of: ≥ .995 ≥ .995 :AA ≥ .995 :Cold Vapor	± 10% of true value	± 15% of true value or EPA limit

Establisment and Utilization of Acceptance Limits

X. DATA REDUCTION, VALIDATION AND REPORTING

Final results are entered into the LDMS system by the analyst, independently reviewed/validated by another analyst or supervisor experienced in the method, and approved by the department manager/lab director. Exhibit 16 describes the flow of samples through the laboratory.

All quality criteria (accuracy, precision, control limits, etc.) are reviewed and approved by the technical staff and independently monitored by the Quality office. Each project is assigned to a project manager. The project manager is responsible for tracking sample progress in the laboratory and ensuring delivery of the product as specified by the client.

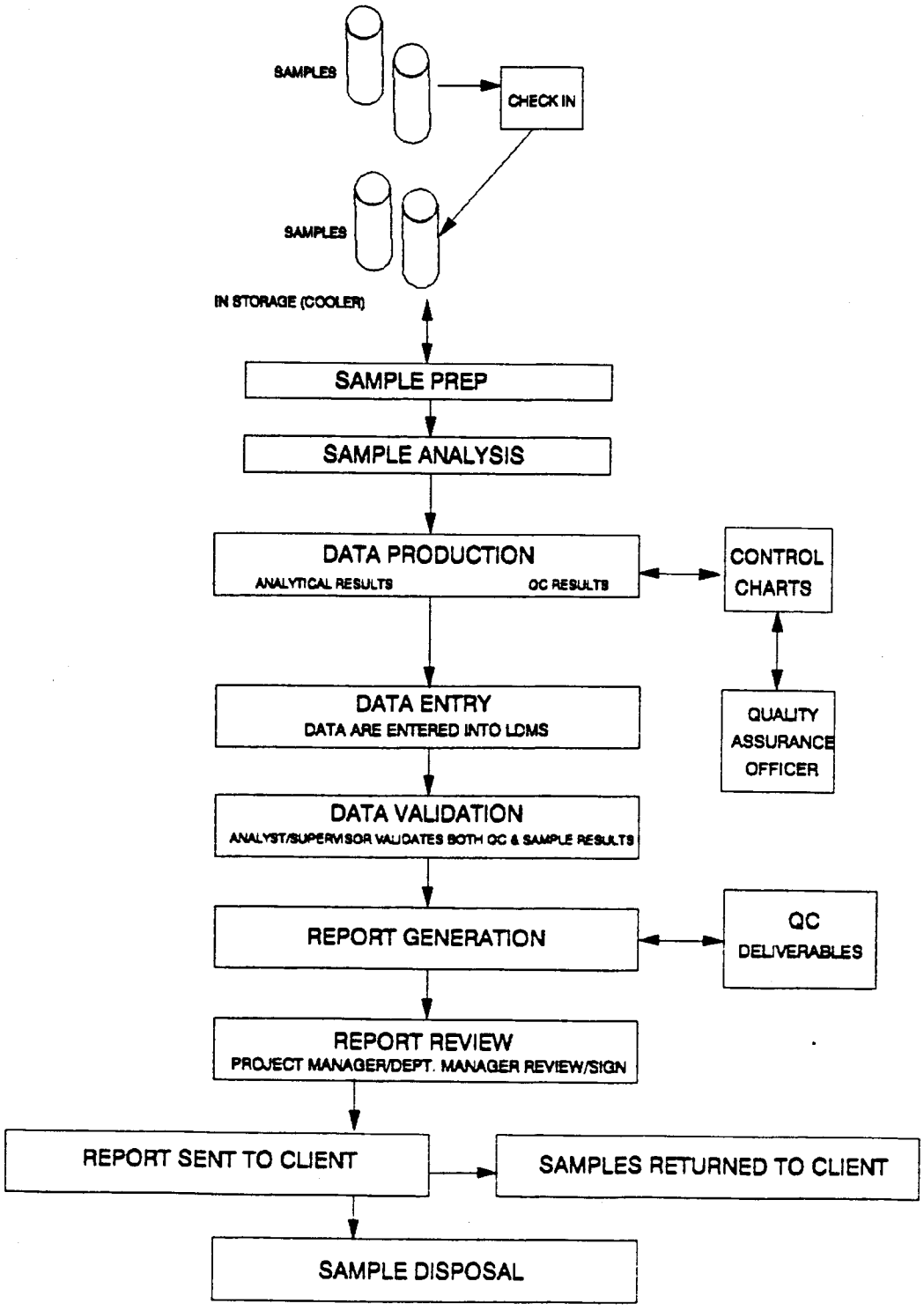
The report is approved and signed by the department manager or director.

Complete project files are periodically inventoried and stored off-site in a secure facility. Electronic data are copied onto computer tape, inventoried and stored off-site in a secure facility.

Sample information flow through the LDMS, and the sequential generation of reports used to manage workloads are illustrated in Exhibit 17.

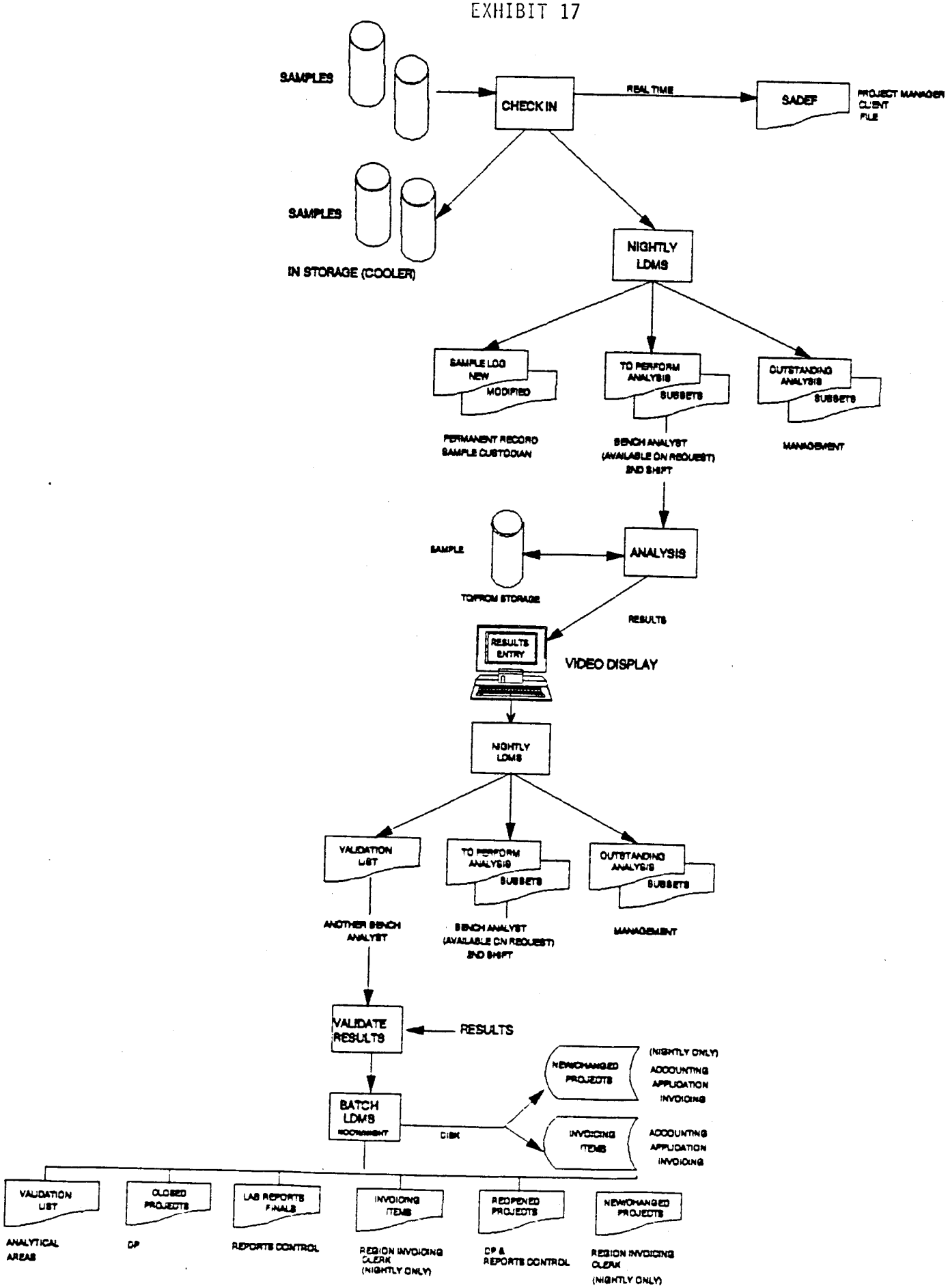
EXHIBIT 16

LABORATORY SAMPLE FLOW SCHEMATIC



INFORMATION FLOW SCHEMATIC

EXHIBIT 17



XI. INTERNAL QUALITY CONTROL

PACE, Inc. quality assurance practices consist of general quality control and assessment procedures that are adapted to the specific operating conditions within each section. The general elements of quality control are outlined below.

A. BLANK ANALYSIS

Reagent: A reagent blank consists of laboratory pure water and any reagents added to a sample during analysis only, or straight solvent.

Method blank: A method blank is a water or soil blank which undergoes all of the preparation procedures applied to a sample (i.e., extraction, digestion).

It is standard policy throughout the laboratory to prepare and analyze a reagent or method blank (whichever is appropriate) with each sample batch. Separate water and soil method blanks are prepared for mixed matrix batches.

Reagent blanks may also be inserted at regular intervals on large batches (of no more than 20 samples), or after highly concentrated samples to check for carryover/contamination. For methods utilizing surrogate compounds, the surrogates are added to all blanks and are subject to meeting acceptance criteria.

A trip blank is submitted for analysis with most samples analyzed for volatile organic compounds. A field blank or procedure blank may also be submitted at the discretion of the client. Field, procedure, and trip blanks are analyzed upon request of the client. Reagent blanks are run daily on each instrument to check the contaminant level.

B. MATRIX SPIKE AND SURROGATE ADDITIONS

Accuracy and matrix biases are monitored using spiked samples and where possible, surrogate additions. It is standard policy throughout the laboratory to prepare and analyze at least one matrix spike for each batch of 20 samples, for each matrix type within the batch, or as specified by state/project requirements.

Surrogate spiking compounds (if available), are added to and analyzed for, with every sample. A measured amount of spike/surrogate concentration is added to the sample before extraction or preparation. Surrogate spiking is utilized for GC and GC/MS analyses only.

C. DUPLICATE SAMPLE ANALYSIS

Precision is assessed by result comparison of a sample prepared and analyzed in duplicate. It is standard policy throughout the laboratory to prepare and analyze at least one duplicate sample for each batch of 20 samples and matrix type within the batch, or as specified by state/project requirements.

D. STANDARDS

The term standard shall apply to any analyte solution of known concentration which is traceable to a certified reference material. This includes calibration standards, spiking solutions, and laboratory control samples. Claims of traceability establish the accuracy of measurements. Therefore, maintaining standard traceability is critical to the achievement of known and defensible data quality.

To establish traceability, all purchased reference materials (neat and stock solutions) are recorded into section-specific standard log books when received.

All entries and PACE standard labels contain a unique PACE ID number, date received, date opened, and expiration date. Log book entries also include the manufacturer's lot number, certified purity, and storage location. Subsequent preparations of stock, intermediate, and working solutions are also recorded in the standard log books. These entries must include all discrete measurements made during a preparation, parent materials, solvent used, and a PACE ID number.

Exhibit 18 illustrates a standard log book entry. Standard Operating Procedure for standards preparation contains further instructions for assigning unique ID numbers, proper syringe technique, shelf life of standards, and good laboratory practices.

Labeling: The standard vial should have a reference label (covered with cellophane tape) with the following information:

1 - Standard
2 - Name of Standard
3 - Prep. Date
4 - Prep. Personnel Initials
5 - Solvent

Certified reference standards from the EPA Repository are used for calibration or laboratory control standards in many organic analyses. Reference standards may also be purchased from approved commercial vendors. Currently approved vendors for organic reference standards are Ultra-Scientific, Supelco, Chem-Service, Inc., and Aldrich Chemical Company, Inc. Inorganic standards are purchased from major scientific supply companies (Fisher, American Scientific, and VWR). Certificates of analyses are requested with each purchase.

E. METHOD DETECTION LIMIT

The method detection limit (MDL) is defined as the minimum substance concentration that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. In general, the protocol described in Appendix B to 40CFR 136 (Federal Register, Vol. 49, No. 209, 10/26/84) is used to establish MDL's.

For GC/MS analyses and organochlorine pesticides by GC/EDC, the MDL has been determined according to EPA Contract Required Detection Limits (CRDL) as established for the Contract Laboratory Program. The MDL's for other organic analyses are set according to industry standards, client requirements, and instrument/method limitations. The MDL is validated using prepared standard solutions analyzed at detection limit concentrations.

Metals and general (wet) chemistry analyses MDL's correspond to instrument detection limits, and are established in the following manner: A standard solution of analyte in laboratory pure water with a concentration of 3-5 times the estimated instrument detection limit is analyzed seven consecutive times. The MDL is set at 3 times the standard deviation of the seven consecutive measurements.

EXHIBIT 18

NEAT STANDARDS:

NAME: Acephate CODE: Acephate-1

OTHER NAMES: Methamidophos

BRAND: Chem Service WARNING: POISON

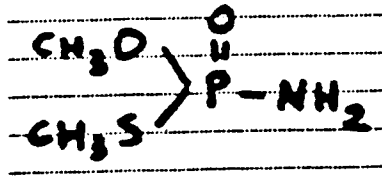
CAT. NO: PS-738

LOT NO: 30-13 LOCATION: Fr. # 2

PURITY: 98% Rack # 1

EXP. DATE: 9-90 position # 23

RECEIV. DATE:



Solvent used: Acetone

Source:

purity:

Lot #:

GC Extraction

DILUTIONS:

code: Acephate-D 1

STOCK SOLUTIONS AND STANDARDS

STD #	CONC. (PPM)	SOLVENT	PREP. DATE	LOCATION
# 502	2,000 ppm	Acetone	3-1-89	Fr. #1, Rack # 5
# 503	D.W. 80 ppm	Hexane	3-2-89	Fr. #1, Rack #7
# 503A	40 ppm	"	"	"
# 503B	60 ppm	"	"	"
# 503C	100 ppm	"	"	"

GC Extraction

For non-instrumental general (wet) chemistry methods, the MDL is established using titrimetric or gravimetric procedures. The MDL for titrimetric procedures is equal to the concentration calculated from a volume of titrant which is one-half of the smallest buret increment. Gravimetric MDLs are calculated from the weight equal to the lowest possible scale reading.

F. CONTROL CHARTS

Control charts monitor daily variations in precision and accuracy of routine analysis and detect variation trends. QC charts are constructed from performance data of the complete analytical method. Control chart construction requires initial data to establish the mean and standard deviation of measurements. Currently, spikes, spike duplicates, RPD's and external check sample values are charted.

G. LABORATORY CONTROL SAMPLES

NIST traceable quality control check samples are analyzed at least quarterly. They provide a means of assessing the accuracy and precision of a measurement system's performance. Parameters of interest that initially fall outside of QC acceptance criteria are compared against a prepared EPA QC check sample. If laboratory performance for the parameter is found to be out of control, then necessary corrective actions are implemented.

XII. PERFORMANCE EVALUATIONS AND SYSTEM AUDITS

A. PACE's SYSTEM AUDITS

Internal Audits:

1. All records, logs, and data files are audited quarterly for completeness, accuracy, and adherence to standard operating procedures by an internal auditing team. Audit team members include the Quality Assurance Officer and any other associated personnel. Several random project files are evaluated for compliance to procedures throughout the analytical process (i.e., from sample receipt through the final report). Supervisors, and lab analysts routinely check all records for the same criteria.

External Audits:

2. PACE is audited as required by regulatory agencies to maintain laboratory certifications, and by various commercial clients with laboratory auditing programs. These audits include audits by USEPA, USATHAMA, AIHA, and other appropriate federal, state and private agencies.

Total Quality System Audit:

3. The Corporate Quality Office performs a yearly on-site audit at each regional facility. The Corporate audit is conducted by the Vice President of Corporate Quality/the Quality Program Specialist. This audit is designed to evaluate all regional office operations and is not limited to only laboratory operations. Audits may either be systems-related or technical in nature, depending on the type of information needed for making quality improvements. An example of one type of form used is shown in Exhibit 21.

B. PERFORMANCE EVALUATIONS:

1. PACE participates in the US EPA semi-annual drinking water (WS Series) and semi-annual wastewater (WP Series) Performance Evaluation Studies (four studies per year).

2. PACE participates in various client-sponsored performance evaluations by analyzing QC samples prepared and submitted by commercial clients in conjunction with their own QA program.

3. Several government proficiency samples are analyzed annually to maintain various laboratory certifications (Exhibit 19 and 20).

4. PACE regional offices are provided blind QC check samples quarterly. These are provided by Corporate Quality as a part of the PACE Interregional Testing Survey, and may also be provided independently by the regional Quality Assurance Officer.

pace INCORPORATED
THE ASSURANCE OF QUALITY

EXHIBIT 19

State Certifications

July 1991

State Certification	Regional Office: NC Ashe	NC Char	CO	FL	IA	KS	MN	NY	No CA	PA	So CA
AL Drinking Water				●							
CA Air							●		●		
CA Pesticides									●		
CA Hazardous Waste						○	●		●		●
CA Wastewater						○			●		●
CA Drinking Water						○			●		
CO Drinking Water			●			○					
CT Solid and Hazardous Waste								●			
CT Drinking Water								●			
CT Wastewater								●			
CT Environmental										●	
DE Drinking Water										●	
FL Environmental				●			●	○			
FL Drinking Water				●							
IA Drinking Water				●	○	○					
IL Drinking Water							○				
IN Drinking Water										●	
KS Drinking Water						●	●	●			
KS Solid and Hazardous Waste						●	●	●			
KS Environmental							●				
KS Wastewater								●			
KY Drinking Water				●							
MA Drinking Water								●			
MD Drinking Water								○		○	
MI Drinking Water							●				
MN Drinking Water (Microbiological)							●				
NC Biotoxicity	●										
NC Drinking Water	●	●		●							
NC Wastewater	●	●		●							
ND Drinking Water						●	●				
NJ Drinking Water								●		●	
NJ Wastewater								●		●	
NY Air								●			
NY Drinking Water							●	●			
NY Solid and Hazardous Waste							●	●			
NY Wastewater							●	●			
PA Drinking Water										●	
RI Drinking Water										●	
RI Solid and Hazardous Waste										●	
RI Wastewater										●	
SC Drinking Water	●	●									
SC Environmental		●		●							
TN UST				●							
TN Drinking Water		●									
VA Drinking Water		●									
VA Wastewater		●									
WI Drinking Water							●				
WI Environmental							●				
WV Drinking Water										●	

● Certified ○ Certification Pending

EXHIBIT 20

National Certifications

July 1991

Certifications/ Accreditations	Regional Office: NC Ashe	NC Char	CO	FL	IA	KS	MN	NY	No CA	PA	So CA
Successfull Participation in U.S.EPA Contract Laboratory Program (CLP)						●	●	●			
U.S.Army Toxic and Hazardous Materials Agency (USATHAMA)							●				
DOE Hazardous Waste Remediation Action Program (HAZWRAP)							●	●			
Naval Energy and Environmental Support Activities (NEESA)							●	●	●		
American Industrial Hygiene Association (AIHA) Laboratory		●	●				●				
National Voluntary Laboratory Accreditation Program (NVLAP)			●	●			●				
Audited by the Missouri River Division of the U.S. Army COE				○			●		●	●	
NIOSH Proficiency in Analytical Testing (PAT) Program		●	●				●				
Analytical Support Laboratory for Minnesota Superfund Projects							●				
Nuclear Materials License			●								

● Certified ○ Certification Pending

EXHIBIT 21

AUDIT INFORMATION
ANALYTICAL OPERATIONS

ANALYTICAL INFORMATION:

Analysis -

Method(s) -

Analyst(s) -

Instrument(s) -

CERTIFICATION INFORMATION:

_____ Yes _____ No (If yes, name of agency) _____

STAFF TRAINING:

Type -

Documentation -

STANDARD OPERATING PROCEDURES:

Hard Copy - _____ Yes _____ No PACE Format - _____ Yes _____ No

Latest Revision Date -

Utilized (how long) -

Copy Provided to Corporate Quality - _____ Yes _____ No

MDL STUDY: _____ Yes _____ No (if yes, provide date) _____

Protocol Used - EPA _____ Other _____(specify)

Documentation - _____ Yes _____ No - Available in lab _____ Filed _____

Approved by - _____ _____
Analyst: Name(s) and Title(s) Date

_____ _____
Quality Assurance Office:

Page 2
Audit Information

ADHERANCE TO SOP:

Without Exception - _____ Yes _____ No - if no, describe specific variance(s) in detail:

_____

_____

_____

QUALITY CONTROL:

Calibration Protocol -

General Procedure (i.e., 3 pt, 5 pt.) _____

_____

_____

Specific

Initial (new) Calibration Procedure - _____

_____

Frequency -

Last Documented New Calibration -

Continuing Calibration Procedure -

Frequency -

Last Documented Continuing Calibration -

EPA Check Standard used - _____ Yes _____ No Frequency _____

Last Documented Use -

Spike Recovery Data:

Frequency -
Documentation -
Criteria -

Duplicates:

Frequency -
Documentation -
Criteria

Blank Analyses:

Frequency -
Documentation -
Criteria -

Quality Control Chart - _____ Yes _____ No Type _____

Latest Entry - _____

Log Book Available - _____ Yes _____ No Maintenance Record _____

Type of Records -
Latest Entry -

External Standards Used - _____ Yes _____ No

Type -
Origin -
Traceability -

Participation in External P.E. Program:

Program -
Frequency -
Latest Participation -
Results Available -

Evaluation of this Analysis Done During On-site Audit -

_____ Yes _____ No Agency: _____

Latest Evaluation -

Describe (in detail) the Data Review Process that is used for QC and sample analytical data. Especially describe the role of analyst, supervisor, manager, director, QAO.

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

Documented - _____ Yes _____ No

Information Provided By: _____
Auditor's Signature _____ Date _____

B. TRAINING AND TECHNICAL REVIEW

PACE considers competent, well-trained personnel to be a key to successful production of valid and reliable data. An extensive training and technical review program is in place at PACE, Inc. It includes:

1. Training Plans

The type of training required for each new or transferred employee is determined individually. A training plan is established to reflect general training needs and to fulfill job requirements.

2. Training Classes

All sections conduct regularly scheduled training sessions specific to their needs.

Audio/visual training programs and open learning texts are available for use by all personnel.

Other laboratory QA and general training classes are offered periodically.

3. Technical Review Program

All employees are subject to technical reviews with their supervisor. The technical review assesses an individual's training progress and technical development and provides an opportunity to redirect the training plan accordingly to comprehensively cover further developmental needs. The schedule for technical reviews is:

a. New hire or transfer to new position/responsibilities: 6 months, 1 year.

b. After 1 year in same position/responsibilities: annually.

4. Support Programs

Attendance at outside seminars, classes, etc., is highly encouraged. PACE participates in many of these throughout the year. In-house seminars are presented by employees for employee bi-monthly meetings. Various topics are covered, including regulatory items and information from attendance at outside seminars. The PACE in-house library contains current periodicals and journals pertinent to the environmental industry and analytical chemistry, in addition to reference books, text books, and regulatory publications.

XIII. PREVENTIVE MAINTENANCE

PACE maintains service contracts for most major analytical equipment including all chromatography instruments, balances, atomic absorption, and inductively coupled plasma instruments. All instruments and equipment receive routine preventive maintenance, which is recorded in instrument specific maintenance logs. Routine maintenance insures that all equipment is operating under optimum conditions, reducing the possibility of instrument malfunction (consequently affecting sample results). An example of an instrument maintenance log is included (Exhibit 22).

EXHIBIT 22

INSTRUMENT MAINTENANCE LOGBOOK FORM

INSTRUMENT I.D.:

DATE	MAINTENANCE ACTIVITY	NAME

XIV ASSESSMENT OF PRECISION, ACCURACY, COMPLETENESS REPRESENTATIVENESS, AND COMPARABILITY

The Quality Control Program at PACE uses precision and accuracy data to determine the acceptability of analytical results. Precision refers to result reproductibility and accuracy measures the degree of difference between observed and true values. One of every 20 analyses performed at PACE is run in duplicate (precision). Also, one of every 20 samples is spiked with a standard to assist in evaluating the accuracy of the method. Once 20 sets of precision or accuracy data have been obtained, a quality control chart is prepared. The Shewhart technique is the statistical method used to construct the charts. These quality control charts provide a quick visual means for monitoring the daily performance of the laboratory. Exhibits 23 and 24 contain generic examples of accuracy and precision charts along with their corresponding data sheets (Exhibits 25 and 26).

A. ACCURACY

The actual test result is compared to the theoretical result of 100% recovery and the percent recovery is calculated.

$$\% \text{ Recovery} = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spike Quantity}} \times 100$$

The percent recovery must fall within specific control limits for the results to be accepted and subsequent data validated. (See Table 2)

B. PRECISION

The results of the duplicate analyses are computed and the absolute relative percent difference (RPD) is calculated.

$$\text{RPD} = \frac{|\text{Sample Result} - \text{Duplicate Result}|}{\text{Average Result}} \times 100$$

The RPD must fall within set control limits for the results to be accepted and subsequent data validated. A one-sided distribution with zero as a target value is typical, given absolute value requirements (CLP).

C. WARNING LIMITS

Warning limits represent the 95% confidence interval and are equal to the mean value for the control sample, plus or minus two standard deviations (± 2S). Exceeding these limits is a warning that the analytical system may be approaching an out-of-control situation, and should be inspected for possible sources of error before continuing the analysis. Analysts will inform the Quality Assurance Officer or the supervisor of such problems.

EXHIBIT 23

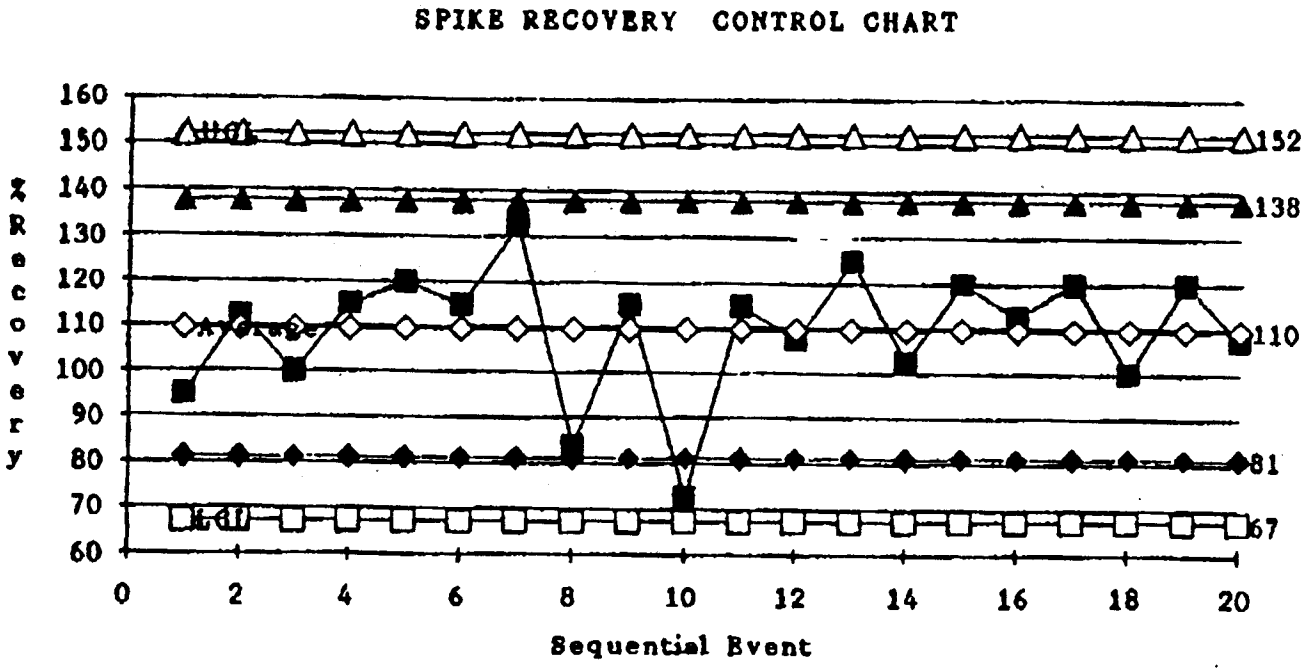
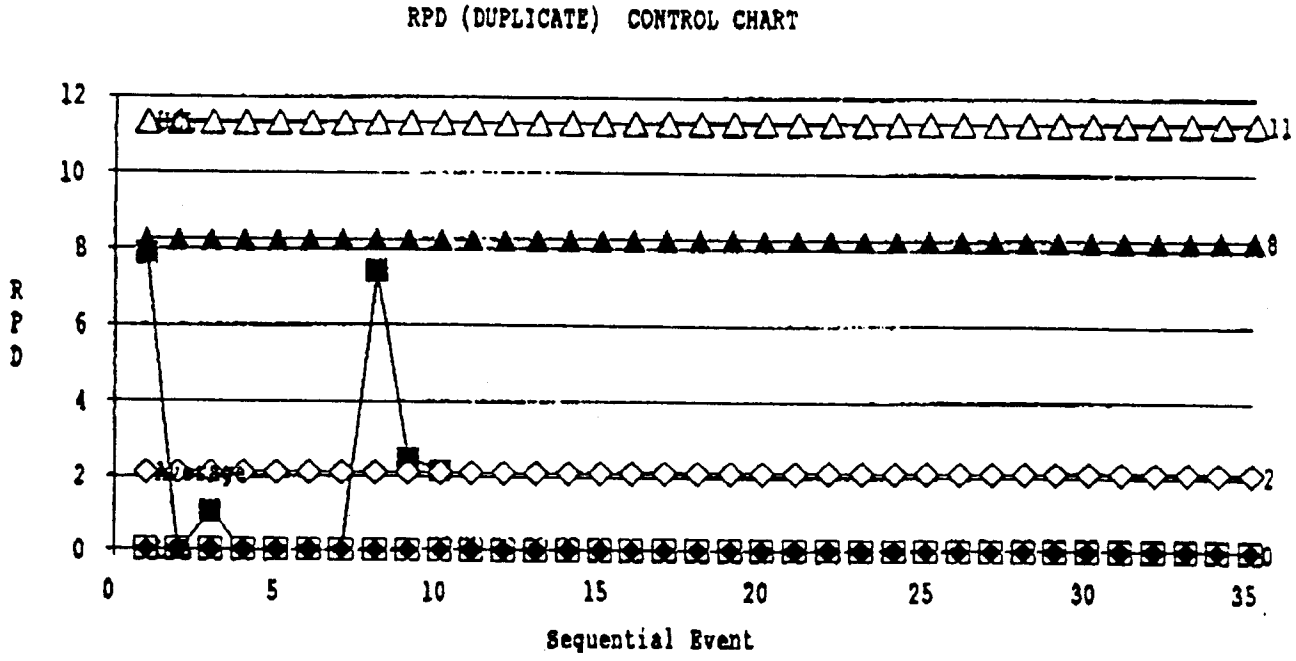

SPIKE RECOVERY CONTROL CHART
% Recovery
160
150
140
130
120
110
100
90
80
70
60
152
138
110
81
67
0
2
4
6
8
10
12
14
16
18
20
Sequential Event

EXHIBIT 24


RPD (DUPLICATE) CONTROL CHART
12
10
8
6
4
2
0
R
P
D
11
8
2
0
0
5
10
15
20
25
30
35
Sequential Event

BENZ-DS.XLS

PACE , Inc.
QUALITY ASSURANCE
SPIKE RECOVERY CHART

Instrume D
Matrix: Groundwater

DATE: ########
TIME: 14:17:40

Compound: [redacted]
Method: EPA0602

	Date	ACTUAL Spike Recovery	THEORY Spike Recovery	[redacted]	LCL Ave-3sd	LWL Ave-2sd	Average Recovery	UWL Ave+2sd	UCL Ave+3sd
2	4/5/91	19.00	20.00	[redacted]	67	81	110	138	152
3	4/5/91	45.00	40.00	[redacted]	67	81	110	138	152
4	4/5/91	20.00	20.00	[redacted]	67	81	110	138	152
5	4/6/91	23.00	20.00	[redacted]	67	81	110	138	152
6	4/7/91	24.00	20.00	[redacted]	67	81	110	138	152
7	4/8/91	23.00	20.00	[redacted]	67	81	110	138	152
8	4/8/91	53.00	40.00	[redacted]	67	81	110	138	152
9	4/9/91	16.80	20.00	[redacted]	67	81	110	138	152
10	4/11/91	23.00	20.00	[redacted]	67	81	110	138	152
11	4/12/91	29.00	40.00	[redacted]	67	81	110	138	152
12	4/18/91	23.00	20.00	[redacted]	67	81	110	138	152
13	4/18/91	43.00	40.00	[redacted]	67	81	110	138	152
14	4/24/91	50.00	40.00	[redacted]	67	81	110	138	152
15	4/25/91	41.00	40.00	[redacted]	67	81	110	138	152
16	4/25/91	24.00	20.00	[redacted]	67	81	110	138	152
17	4/26/91	45.00	40.00	[redacted]	67	81	110	138	152
18	4/29/91	24.00	20.00	[redacted]	67	81	110	138	152
19	4/30/91	20.00	20.00	[redacted]	67	81	110	138	152
20	5/1/91	48.00	40.00	[redacted]	67	81	110	138	152
1	5/2/91	43.00	40.00	[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152
				[redacted]	67	81	110	138	152

Ave Recovery	[redacted]
Std Deviation	[redacted]

EXHIBIT 26

NO3-NICL.XLS

PACE, Inc.
QUALITY ASSURANCE
PRECISION CONTROL CHART

Matrix: Water

DATE:	########
TIME:	14:35:37

Compound: NO3-N-IC-Low

Method: EPA#300.0

Low=1-100mg/l

MDL=1mg/l

	Date	DUPLICATES X1	X2	Relative Percent Difference (RPD)	LCL Ave-3sd (Min=0)	LWL Ave-2sd (Min=0)	Average RPD	UWL Ave+2sd	UCL Ave+3sd
2	2/22/91	1.10	1.19	[illegible]	0	0	2	8	11
3	3/6/91	0.80	0.80	[illegible]	0	0	2	8	11
4	3/7/91	0.98	0.99	[illegible]	0	0	2	8	11
5	3/13/91	1.05	1.05	[illegible]	0	0	2	8	11
6	4/21/91	3.50	3.50	[illegible]	0	0	2	8	11
7	4/24/91	10.60	10.60	[illegible]	0	0	2	8	11
8	4/27/91	2.10	2.10	[illegible]	0	0	2	8	11
9	4/28/91	1.40	1.30	[illegible]	0	0	2	8	11
10	5/3/91	4.10	4.00	[illegible]	0	0	2	8	11
11	5/15/91	9.30	9.10	[illegible]	0	0	2	8	11
12					0	0	2	8	11
13					0	0	2	8	11
14					0	0	2	8	11
15					0	0	2	8	11
16					0	0	2	8	11
17					0	0	2	8	11
18					0	0	2	8	11
19					0	0	2	8	11
20					0	0	2	8	11
1					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11

Average RPD	[illegible]
Std Deviation	[illegible]

D. CONTROL LIMITS

Control limits represent the 99% confidence interval and are equal to the mean value of the control sample, plus or minus three standard deviations (±3S). Exceeding these limits indicates that the analytical system is out-of-control. The Quality Assurance Officer or the supervisor shall be informed and corrective action shall be taken.

1. Method of Setting Limits

Control limits are established via statistical analysis, using QC sample results. Limits are determined for a parameter of each method as analyzed on a specific instrument.

The mean value (P) and the standard deviation (S) for each data set is calculated and the limits are set as:

Warning (WL) = P + 2S = 95% Confidence limit
Control (CL) = P + 3S = 99% Confidence limit

Where $P = \frac{X1 + X2 + X3.... Xn}{n}$ x = Sample result

and $S = \frac{\sum(X - P)^2}{n-1}$ n = Total # of results in set
P = mean value

The minimum number of results to be used for statistical calculation (n) is 15-20. Limits will generally be calculated from a data point set every 30 days, depending on the method. Updated limits are issued at the beginning of every month.

2. Utilization of Acceptance Limits

QC sample results must fall within the established warning limits (P ± 2S) for each parameter.

Results that fall outside of warning limits, but remain within the control limits (P ± 3S), are consider suspect. These results must be carefully examined for possible sources of error in the analysis, or justified as a matrix bias effect. All such results are recorded in a Discrepancy Report form/Corrective Action form (See section XV).

Any three consecutive results outside of warning limits but within control limits is an out-of-control event which shall be documented and corrected.

Results that fall outside of control limits (P ± 3S) must be documented and corrective action taken.

Six consecutive points on the same side of an established mean indicate a trend and require corrective action.

E. COMPLETENESS

Data completeness can be quantified during data assessment. It is expected that laboratories should provide data, meeting QC acceptance criteria, for 95% or more of the requested determinations. It is incumbent for planners to identify any sample types, such as control or background locations, which require 100% completeness.

F. REPRESENTATIVENESS

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in each work plan.

G. COMPARABILITY

Comparability is also considered during preparation of the work plan. The objective of comparability is to ensure that results of similar activities conducted by different parties are comparable. For example, the use of EPA-approved, etc., methods and procedures ensure comparability with other data from previous or following studies.

XV. CORRECTIVE ACTION

If, as a result of audits or QC sample analyses, methods systems prove to be unsatisfactory, corrective action shall be implemented. The project manager, department manager, Quality Assurance Officer, supervisor, and analyst may be involved in the corrective action. If previously reported data are affected by a situation requiring correction or if the corrective action impacts a project budget or schedule, the action will directly involve the project manager (and Quality Assurance Officer).

For immediate or long-term corrective actions, steps comprising a closed-loop corrective action system are as follows:

1. Define the problem.

2. Assign responsibilities for problem investigation.

3. Investigate and determine the cause of the problem.

   a. Check all calculations

   b. Re-analyze the sample

   c. Verify the integrity of the spiking solution, laboratory control sample, or calibration standard.

   d. Check instrument and operating conditions to preclude the possibility of malfunctions or operator error.

4. Determine the corrective action(s) necessary to eliminate the problem.

5. Assign and accept responsibilities for implementing the corrective action.

6. Establish the effectiveness of the corrective action and implement the correction.

7. Verify and document that the corrective action has eliminated the problem (using a Discrepancy Report form)

   Depending upon the nature of a problem, the corrective action implemented may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented.

   In addition, if the corrective action mandates the preparation of a new standard or calibration solution(s), a comparison study between the new solution versus the old solution will be performed. The results are supplied with the weekly QC submittal as verification of problem elimination.

XVI. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quarterly reports are provided by the Quality Assurance officer to the President, Vice President of Quality and Regional Director. This report addresses the quarterly quality assurance activities including details of corrective actions implemented, audit results, and QC summary information.

REFERENCES

1. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, U.S. EPA 600/4-79-019, March, 1979.

2. Federal Register, 40 CFR Part 136, October 26, 1984.

3. Test Methods for Evaluating Solid Waste, U.S. EPA SW-846, September, 1986.

4. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.

5. Standard Methods for the Examination of Water and Wastewater, APHA, AWWA, WPCF: 16th Edition, 1985.

6. NIOSH Manual of Analytical Methods, U.S. Department of Health, Education, and Welfare; Second Edition, 1977.

7. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079-C.

8. Methods for Chemical Analysis of Water, Wastes, EPA-600/4-79--020, 1983.

9. California Administration Code, Title 2, Chapter 30, Article II, "Criteria for Identification of Hazardous and Extremely Hazardous Wastes."

10. The Determination of Inorganic Anions in Water by Ion Chromotography - Method 300.0 Test Method, EPA-600/4-84-017. March, 1984.

11. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 2nd Edition, U.S. EPA, revised April, 1984.